

Phase I Remedial Investigation Pasco Landfill Pasco, Washington

Volume II - Sampling and Analysis Plan Appendices

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Prepared for:

Pasco Landfill PLP Group

Project 624419

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APPENDIX A

Laboratory Quality Assurance/Quality Control (QA/QC) Plans



SOUND ANALYTICAL SERVICES, INC.

QUALITY ASSURANCE PROGRAM PLAN

Quality Assurance Program Plan for Sound Analytical Services, Inc.

Document Control #QAAP=01

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1.0 Introduction

This Quality Assurance Program Plan (QAPP) describes the methods and procedures that will be used by Sound Analytical Services (SAS) to assure the quality, precision, accuracy and completeness of the data generated in the laboratory.

This QAPP has been prepared to provide assurance that the work performed by SAS will be of the sufficient quality to meet data quality objectives and to be responsive to the requirements of the client.

This QAPP is based on the Interim Guidelines and Specifications for preparing Quality Assurance Program Plans, QAMS-005/80 (US EPA February, 1983), QA Project Plan Guidelines by Washington State Dept. of Ecology, May, 1991 and Guidelines and Specifications for Preparing Quality Assurance Program Plans (QAMS-004/80), September 20, 1980.

The QAPP focuses on the acquisition of environmental data of known and acceptable quality. Other aspects of the program, such as report preparation and records, will be controlled by the internal requirements of SAS's Quality Assurance Program and the SAS Quality Assurance Manual. The policies and procedures specified in this manual define acceptable practices applicable to most environmentally related projects, regardless of the specific aim of the project.

1.1 Program Objectives

The objective is to secure an analytical laboratory which produces reliable results with a prompt turnaround time in order to facilitate services at sites that are of environmental concern.

2.0 Quality Assurance Policy Statement

Sound Analytical Services, Inc., is committed to providing quality services for EPA, state and local regulatory requirements while meeting the diverse analytical environmental service needs of our clients.

To satisfy our clients' data quality objectives, to meet regulatory requirements and to comply with SAS management requirements, SAS uses approved analytical methodologies and state of the art laboratory techniques. SAS has adopted a comprehensive Quality Assurance Program. The principles and practices of the Program apply to every employee at every level within SAS; they are fundamental to the way we do business and to the services we provide.

The SAS Quality Assurance Manual defines the overall statement of Program policy for the SAS laboratory. The SAS QA manual along with established Standard Operating Procedures provide guidance for fulfilling our quality assurance objectives as well as a statement of the laboratory's commitment to deliver a high quality service.

Implementation of the Program is the responsibility of all SAS laboratory personnel. Management at every level has the duty and authority to insist that these responsibilities are met and that the principles and practices of the Program are fulfilled. The Quality Control Coordinator (QCC) assures that the Laboratory Quality Assurance Program operates on a day-to-day basis as intended. The QCC has the authority and duty to stop work if systems are not in compliance and when necessary to satisfy Program requirements.

To verify that the Quality Assurance Program is successfully implemented, the laboratory is independently audited by State and client auditors. The laboratory is internally audited on a regular basis by the QCC.

An SAS QA/QC manual is available upon request.

Dennis L. Bean Date

Laboratory Director, SAS

Christish Sheef Date 7/23/92

J. Christopher Shaeffeld Quality Control Coordinator, SAS

3.0 Quality Assurance Management

In order for the program to be successful, the laboratory must provide three major segments to the program. These include:

- 1. Communication and planning with the client
- 2. Successful performance of analyses within the required time limits
- 3. Supporting QA/QC data to validate the analytical data

All three must be accomplished conjunctively for the success of the program.

3.1 Communication

The communication between the Client and the SAS laboratory must be accurate and correct due to turnaround time constraints. In order to ensure efficient communication, SAS will designate a member of the laboratory management as a coordinator for each project. The coordinator will be responsible for discussing analytical requirements, delivery times and reporting requirements on a case by case basis. This dialogue is necessary to meet the requirements of the project.

3.2 Sample Flow and Analysis

Upon receipt of the samples, each sample delivery group is logged into the laboratory log book. Each sample is assigned a unique number and is designated for the requested analysis. The laboratory group leaders are apprised immediately of sample receipt and testing criteria. The group leader is responsible for the performance of the analysis according to the methodology established in the QAPP. After analysis, the group leader validates the analysis and forwards data to the QC Coordinator The QC Coordinator completes the review and data is returned to sample control for word processing and draft report generation. After further review a final report is generated for the client. In all instances, analyses and the review process will be completed within the TAT limit.

In general, the laboratory functions must be efficient to produce quality analytical results within the limited turnaround. The performance of the analyses for routine parameters and characterized waste are straight forward and present little problem to the environmental laboratory. The analysis of unknown waste may present a more complex situation which requires special sample handling and /or analysis. In either case the laboratory must be competent and precise in its' technology.

3.3 QA/QC

The development of an acceptable and accurate QA/QC plan is extremely important to the success of the project. This plan must discuss not only analytical but also operational compliance with the analytical methodology as stated in the methodology. Modifications to the methodology must be communicated to the client.

Quality assurance/control encourages a number of laboratory functions to ensure a quality product. These include:

Organization- This includes the analyst, QA/QC Coordinator and upper management.

<u>Documentation</u> SAS stands by its QA/QC document as well as its SOPs which contain method specific QC procedures and limits.

Training- All analysts must be trained and that training be documented and filed.

<u>Analyses</u>- All analyses must be acceptable to regulatory agencies.

<u>Materials Procurement</u>- All materials used in the laboratory must be of a grade to provide accurate analyses.

4.0 Personnel Qualifications and Organizational Structure

Brent A. Hepner is the president of Sound Analytical Services. Mr. Hepner has over 12 years of experience in the analytical laboratory field. As president and majority owner of SAS, Mr. Hepner was involved in designing the laboratory to meet the requirements of the analytical procedures and instrumentation. As director of the pesticides section he has developed the quality control program and Standard Operating Procedures that are currently in use.

Thomas R. Boyden serves as the vice-president and laboratory manager and brings over 13 years of experience in the analytical laboratory field. Mr. Boyden was involved in the design phase of SAS' new laboratory facility. He is responsible for authoring and implementing the overall Quality Assurance Program for the laboratory.

Stan P. Palmquist serves as secretary-treasurer and trace metals director and has more than 13 years of experience in the laboratory field. At Sound Analytical his responsibilities include the operation of the Leeman Labs Inductively Coupled Plasma Spectrophotometers and the Thermo-Jarrell Ash Smith-Hieftje Graphite Furnace AA for the analysis of trace metals in environmental samples.

Dennis L. Bean is the laboratory director and director of the GC/MS section. Mr. Bean brings with him 9 years of GC/MS experience obtained at Hazleton Laboratories of America and as an applications chemist for Finnigan MAT, Inc., a major manufacturer of GC/MS systems. His experience includes instrument troubleshooting, GC/MS applications programming, GC/MS method development and validation, GC/MS analysis in accordance with EPA-CLP requirements and analysis of a wide range of pesticides using GC-MS.

J. Christopher Shaeffer is the QC coordinator and brings with him over 20 years of experience in the analytical laboratory field. His experience as QC coordinator at IT Corp. and as laboratory director for the Edwards Aquifer Center in Texas equips him to closely coordinate QC within SAS and to assure compliance with Federal and State requirements. In addition, Mr. Shaeffer is responsible for overseeing SAS's certification status and acts as the laboratory safety officer.

Lila Accra Transue is a Project and Account Manager for SAS. She is our main line of communication on larger projects and her extensive technical and educational background in the environmental industry enables us to assure client and regulatory satisfaction with our services. Ms. Transue is an environmental chemist with over eight years of environmental, analytical and field laboratory experience and knowledge. She worked for the Washington State Department of Ecology and most recently was the senior corporate chemist for Ecology and Environment, Inc./Seattle Region.

Marty French is an organic chemist and Group Leader in charge of fuels analysis at SAS. Mr. French brings 4 years of experience in the lab from U.S. Oil & Refining Co. His duties at SAS include operation, maintenance and scheduling of gas chromatographic systems used in the analysis of soils and waters for gasoline, BTEX, diesel and heavier petroleum compounds.

<u>C. Larry Zuraw</u> is the lead chemist performing volatile organic analyses in the GC/MS section. Mr. Zuraw draws on over 23 years experience in the analytical lab. His duties include the GC/MS analysis of fuel hydrocarbons, volatile halocarbons and aromatics in soil, water and air.

Dean Strom is a chemist in the fuels analysis laboratory. His main responsibility is for the analysis of extractable hydrocarbons by EPA Method 8015 and Washington State DOE Method WTPH-D. He is also proficient in the analysis of volatile fuel hydrocarbons.

<u>Dawn Werner</u> is a lab technician in the fuels analysis lab. Her duties include the analysis of petroleum hydrocarbons using EPA Method 418.1 and Washington State DOE Method WTPH-418.1

Modified. She also assists in the extraction and analysis of diesel range hydrocarbons in soil and water.

<u>Peter Baccetti</u> is responsible for the analysis of environmental and drinking water samples for trace metals. His duties include the operation of both the sequential and simultaneous ICP instruments and the graphite furnace AA.

Randall Kueter has previous experience as a wastewater treatment plant operator in the city of Yankton, SD, where he performed EPA-required biological and chemical analysis per Standard Methods procedures. His responsibilities at SAS include conventional analysis of water and wastewater using the Alpkem Rapid Flow Analyzer and wet chemistry procedures.

Robert Baccetti is a laboratory technician responsible for scheduling and performing all aspects of sample weighing and preparation for trace metals and trace organics analysis.

Andrew Wahto is a laboratory technician with over four years experience in the preparation and analysis of transformer oils for PCB's by gas chromatography. in addition, he has five years of experience in the analysis of fats and oils and other feed products.

<u>Eric Phillips</u> is a laboratory technician whose primary function is the operation of an INCOS 50 GC-MS in the analysis of sample extracts for semi-volatile organic parameters. Prior to that he prepared samples for GC-MS analysis using EPA methods. He is also experienced in mercury analysis using cold vapor AA and in TCLP extraction procedures.

Guy Archibald is a laboratory technician experienced in the preparation of soils and other solid waste for trace metals analysis using microwave digestion techniques. His experience also includes the conventional analysis of water and wastewater and the analysis of meat, fishmeal products, and fats & oils.

Alfonso Benitez is a laboratory technician whose main duty is to prepare samples for volatile organic analyses. As a lab technician at RETEC, Inc., he became proficient at organic extractions for PAH's, phenols, O&G's and TPH's.

A complete organizational chart is provided in Appendix A.

5.0 Facilities, Equipment and Services

Sound Analytical Services currently occupies a 14,500 sq.ft. facility in Fife, Washington. It is a full service environmental testing laboratory which began operations in November, 1985.

The facility is divided into eight discrete sections to minimize cross contamination. Approximately 2,200 sq. ft. is dedicated to general laboratory space, 2,000 sq.ft. to volatile organics, 2,000 sq.ft. to semi-volatile organics, 650 sq. ft. to PCB, TOX and TOC analysis, 650 sq.ft. for trace metal analysis, 1000 sq.ft. for sample management, 3000 sq.ft. for administrative office space and 2600 sq.ft. for storage and utility space.

5.1 Instrumentation

Includes 4 Gas Chromatograph/Mass Spectrophotometers, 11 Gas Chromatographs with various detectors, 2 Inductively Coupled Plasma Spectrophotometers, 1 graphite furnace AA, 1 fully automated cold vapor mercury analyzer, 2 rapid flow autoanalyzers for general chemistry, 1 Beckman IR, 1 Dohrmann TOC analyzer, and 1 Dohrmann TOX analyzer. Each of these systems is operated by a computer system.

5.2 Staff

The staff consists of 21 chemists/analysts, 4 technicians and 7 administrative and clerical personnel.

6.0 Data Generation

As stated, the overall objective of the Quality Assurance Program for the SAS laboratory is to provide data of known quality. To accomplish this, the laboratory must:

Maintain an effective, ongoing Quality Control Program to measure and verify laboratory performance.

Meet data requirements for accuracy, precision, and completeness through the use of proven methodologies.

Provide sufficient flexibility to allow controlled changes in routine methodology to meet specific data requirements.

Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed.

Recognize and promptly correct for any factors which adversely affect quality.

Maintain complete records of sample submittal, laboratory performance and completed analyses to verify resulting data. Specifically, the following actions are taken:

Procurement and control of instrumentation and supplies required for laboratory operation

Sample receipt, chain of custody completion and sample storage.

Calibration and preventive maintenance of instrumentation.

Establish a Quality Control sample program within the laboratory so that statistical evaluation of analyses can be performed.

All analyses are performed in accordance with recognized analytical procedures.

Data processing, validation and reporting.

Control and maintenance of laboratory records.

Identification and resolution of nonconformances requiring corrective action.

Audits to verify laboratory performance and the reporting of audit results to management.

Training of analysts in technical and quality control procedures, including an orientation to the SAS Quality Assurance Program.

6.1 CHAIN OF CUSTODY

An overriding consideration for resulting data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody and disposal must be documented to accomplish this. Documentation is accomplished through the chain of custody record that lists each sample and the individuals responsible for sample collection, shipment and receipt. A sample is considered in custody if it is:

In a person's actual possession

In view after being in physical possession

Locked or sealed so that no one can tamper with it after having been in physical custody.

In a secured area, restricted to authorized personnel.

Appendix B shows the chain of custody form used by Sound Analytical Services for collecting and shipping samples. SAS will not accept samples collected by SAS personnel for analysis without a correctly prepared chain of custody.

The chain of custody form will be signed by each individual who has had the samples in their possession. Preparation of the chain of custody will be as follows:

The chain of custody record will be initiated in the field by the person collecting the sample. Every sample will be entered and given a unique number on the COC. Samples can be grouped for shipment and use a common form and if there are more samples than positions on the form another form should be initiated. The record shall be completed in the field to indicate project, sampling team, etc.

If the person collecting the samples does not transport the samples to the laboratory or deliver the sample containers for shipment, the first block for relinquished by ______, received by ______, will be completed in the field.

The person transporting the samples to the laboratory or delivering them for shipment will sign the record form as relinquished by_____.

If the samples are to be shipped to the laboratory by commercial carrier, the chain of custody form will be sealed in a water tight container, placed in the shipping container and the shipping container sealed prior to giving it to the carrier.

If the samples are directly transported to the laboratory, the chain of custody form will be kept in the possession of the person delivering the samples.

For samples shipped by commercial carrier, the waybill will serve as an extension of the chain of custody record between the final field custodian and receipt in the laboratory.

Upon receipt in the laboratory, the sample custodian will open the shipping containers, compare the contents with the chain of custody form, and sign and date the record. Any discrepancies will be noted on the chain of custody form.

If any discrepancies occur, the samples in question will be segregated from normal sample storage and the client will be immediately notified.

The chain of custody is completed after sample disposal.

Chain of custody records will be maintained with the file for a specific project, becoming part of the data package.

Multiple part chain of custodies are used so that a copy can be kept by each person who has custody of the samples.

7.0 Data Processing

Data validation (review) is the process whereby data are screened and accepted or rejected, based on a set of criteria. Data will have been provided by trained analysts using approved methods and instrument systems in control.

Data validation is a systematic procedure of reviewing a body of data against a set of criteria to verify its validity prior to its intended use. It is applied to a body of data after the fact, systematically and uniformly. It must be applied close to the origin of the data by an independent and objective reviewer.

validation begins with the processing of Data data continues through review of the data and the reporting of the analytical results. Data processing can be performed by the analyst who obtained the data or another analyst. Data review starts with the analyst independent of the data acquisition and processing, or the group leader, who assures that the data processing has been correctly performed and that the reported analytical results correspond to the data acquired processed. Checks are made for internal consistency, proper identification, transmittal errors, calculation errors transcription errors. Final review of the data to be reported is performed by the Laboratory Manager.

As stated, the first step in validation is data processing. In general, data will be processed by an analyst in one of the following ways:

Manual computation of results directly on the data sheet or on the calculation pages attached to the data sheets

Input of raw data for computer processing

Direct acquisition and processing of raw data by a computer

If data are manually processed by an analyst, all steps in the computation will be provided including equations used and the source of input parameters such as response factors, dilution factors and calibration constants. The analyst will sign or initial and date in ink each page of calculations.

For data that are entered by the analyst and processed using a computer, a copy of the input will be kept and uniquely identified with the project numbers and other information as needed. The samples analyzed will be evident and the input signed and dated by the analyst.

If the data are directly acquired from instrumentation and processed, the analyst will verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The analyst will sign and date the resulting output.

8.0 Data Quality Assessment

The following is a discussion of two methods to be used for reviewing (checking) data processing. At least 20 percent of all data will be checked using one of these procedures. If during the checking process, errors are determined in a data set, checking will be completely (100 percent) performed for the data set.

8.1 Checkprint Procedure

The analyst performing the data processing will present the data package to an analyst independent of the work. The package will include as appropriate; raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors, etc.

The independent analyst (checker) will make and review copies of the data for:

Appropriateness of the equations used

Correctness of numerical input

Numerical correctness of all calculations; this will be done by recalculating numerical computations

Correct interpretation of strip charts, etc.

All entries and calculations that the checker reviews will be marked in ink with a check mark or highlighted on the copies. The checking process must be thorough enough to validate the results as correct. If the checker disagrees with any part of the computations, the checker will mark through the number with a single line and place the revised number above it.

Any changes made by the checker will be backchecked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so that they agree with the result presented.

The checker will sign or initial originals and date in ink pages of data that were reviewed (except for groups of

printout such as chromatograms). Signing or initialling and dating indicates that the reviewer agrees with the calculations and that any changes made have been agreed to by the originator and entered on the originals.

If the data have been processed by computer, the reviewer will check at least 20 percent of input entries. Agreement will be indicated by a check mark for every line. If the checker disagrees with the input the number should be marked through with a line and the correction indicated above it. Corrections must be backchecked by the originator.

If an input error is identified and the data have been processed, it will be necessary to reprocess the data. In this event, the checker will mark the second set of input to indicate agreement with the input changes. The checker will sign and date in ink the computer input to indicate agreement.

Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.

Use of the copies of data for checking produces a "checkprint" which will be maintained as a record to document the review performed.

8.2 Original Check Procedure

The "original check" procedure is an alternative to the checkprint process; instead of a copy (checkprint) being used, analysts:

Mark on originals for checking in black ink, initialling and dating these entries as above, backcheck changes in black ink using lineouts, initials and dates.

Summary checklists may be used and attached to the checked data which clearly describes the data checked, what the check was for, who did it and when and if the data were acceptable.

Nonconformances and corrective actions such as reprocessing must be documented.

8.3 Review of Data Reports

Review of data reports is required to verify that information reported by Sound Analytical Services corresponds with processed analytical results. Review is only required of the data as it is presented for issuance. Intermediate steps

performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation.

After the draft data report is prepared, generally in tabular form, the reported results should be checked against the reviewed processed data (not subsequent summaries) so that transcription errors do not occur. The checking process is as follows:

Using the draft report, all data entries are checked. The checker can be an analyst or group leader. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.

The draft data report should be checked so that the items cited for data presentation are complete and correct. As the reviewer checks the entries on the draft report, an ink check mark is placed beside each correct entry. Corrected entries are marked through with a single line and the correct entry provided; changes are documented with initials and date. The reviewer will indicate that corrections have been made in the report by placing a second check mark at the correction after comparing the change with the revised copy.

Use of the draft data report results in a checkprint which should be maintained as a record to demonstrate the review

If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout.

If the computer output is used directly as the data report without further transcription, only the input requires review.

After checking of the data report is complete, it is given to the QC Coordinator or the Project Manager for final review. This step is not intended to verify the reported data but rather to determine that the report meets project requirements. From the QC Coordinator, the data report is given to the Laboratory Manager for final approval to issue.

8.4 Data Reports

The format and content of a data report are dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements are met, etc. Sound Analytical Services Quality Assurance Program does not specify a report format; however the following are applicable to data presentation:

The final data presentation will be checked and verified by the previously stated program and approved by the Laboratory Manager

Data are presented in a tabular format whenever possible

Data are generally formatted as a Certificate of Analysis or as a memorandum

Each page of data is identified with the project number and name; date of issue; and, if appropriate, client name

Data presentation includes:

Sample identification number used by SAS and the sample identification provided by the client.

Chemical parameters analyzed, reported values, units of measurement and the analytical method used for the types of analysis specified.

Detection limit of the analytical procedure if less than the detection limit is reported.

Data for a chemical parameter reported with consistent significant figures for all samples

Results of Quality Control samples analysis if appropriate

Achieved accuracy, precision, and completeness of data if appropriate

Footnotes referenced to specific data, if required, to explain reported values

Data should be released from the laboratory only by the Laboratory Manager or Laboratory Director.

Any analytical results verbally communicated are considered preliminary until data are presented in hard copy, in a formal report.

9.0 Nonconformance and Corrective Action

A deficiency is an unauthorized deviation from documented procedures, practices, or standards, or a defect in an item, that could lead to degradation of quality.

A nonconformance is a deficiency in characterization, documentation, or procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. Nonconformances may be caused by non-laboratory

operations, e.g.; field collection paperwork not complete or improper sample containers used.

Nonconformances may include (but are not limited to) the following:

Failure of an instrument to work properly

Sample receiving documentation not correct

Sample condition on receipt not acceptable

Sample holding time exceeded

Sample storage conditions outside criteria

Incomplete sample preparation/analysis procedures used

QC sample data (blank, spike, duplicate, surrogates, etc.) outside limits

Calibration requirements not met

Data recording errors, transcription errors, or failure to document

Data validation errors

Relative standard deviation for response factors greater than accepted limits

Any situation or result which might affect the quality of data

A nonconformance memorandum (see Appendix C) is formal documentation of a nonconformance that includes a description of the problem, the corrective action taken, the individual recognizing the problem and the date discovered, the sample analyses affected (if any) and the initials of appropriate reviewers and the Quality Control Coordinator.

A corrective action is an appropriate measure applied to correct a deficiency and minimize the possibility of recurrence.

Corrective action will include, but not necessarily be limited to:

Recalibration of instrument, using freshly prepared calibration standards

Reanalysis of samples

Replacement of specific lots of solvent or other reagents that produced unacceptable blank results

Additional training of laboratory personnel in correct implementation of sample preparation and analytical methods

Reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements

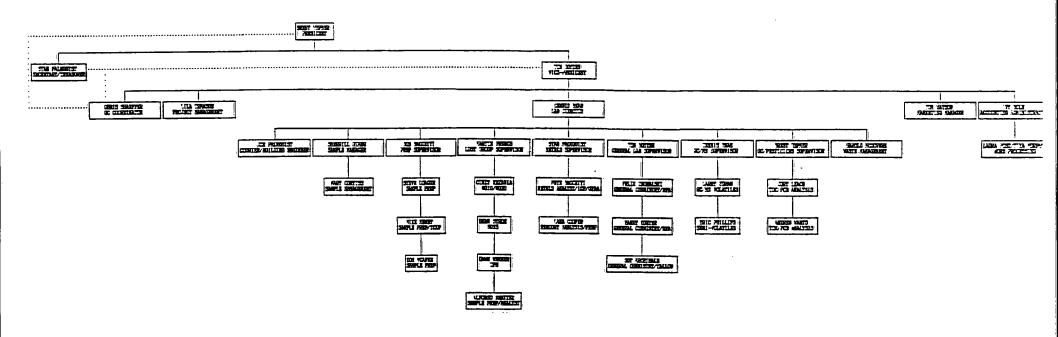
Communication with the clients to determine the appropriate action (e.g.; insufficient sample remaining for reanalysis).

10.0 Responsibilities

All employees of the laboratory staff are responsible for reporting nonconformances they observe/identify.

Employees identifying problems that might affect quality are responsible for reporting them to the appropriate supervisor and signing nonconformance memos.

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SOUND ANALYTICAL SERVICES, INC.

ANALYTICAL & ENVIRONMENTAL CHEMISTS

4813 Pacific Hwy. East Tacoma, Washington 98424 (206) 922-2310 • FAX (206) 922-5047

CHAIN OF CUSTODY / REQUEST FOR LABORATORY ANALYSIS

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SOUND ANALYTICAL SERVICES, INC.

LABORATORY NONCONFORMANCE MEMO

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	COUNTING DATA VERIF.
NONCONFORMANCE [> appropriate item(a)]:	
. O Not enough sample received for proper analysis.	6. 🔲 incorrect preparation/analysis procedure(s) use
g. Q. Sample received	no re-prep or re-analysis possible.
G Broken/Leaking	7. 🖸 Samples analyzed and reported under an inval
☐ Without proper preservative	calibration curve.
🗀 In Improper container	8. 🔲 Incorrect/incomplete data reported to client.
With incomplete paperwork.	9. 🗖 Blank contamination; no re-prep of associate
. 🗖 Holding time exceeded	samples done or possible.
☐ At receipt	10. 🔲 Reported detection limits higher than specifie
☐ In the lab by days.	in:
. 🔾 Sample lost during extraction/analysis; no re-prep	☐ Method ☐ Contract
or re-analysis possible.	QAPP, due to:
QC data reported to client outside of:	Matrix Insufficient sample
☐ Method ☐ Internal	☐ Instrumentation ☐ Other
QAPP Q Contract	_
Regulatory limits.	11. Other (specify):
Associated samples affected:	
CORRECTIVE ACTION:	
Client informed verbally on	by
Client informed in writing on	by
Oneth morning on	• • • • • • • • • • • • • • • • • • • •
Sample(s) on hold until If rel	leased, notify:
Further action required (see back page)	
Corrective action initiated/performed by	Date
Operations Concurrence:	Date
QC Concurrence Nonconformance Deficiency:	Date
CORRECTIVE ACTION VERIFICATION:	
☐ Verified ☐ Cannot verify. Reason:	
	A
NCM Closure: QC Coordinator	Date
NCM Closure: QC Coordinator	Date



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ANALYTICAL SERVICES QUALITY ASSURANCE PROJECT PLAN

FOR

ANALYSIS OF POLYCHLORINATED DIOXINS/FURANS BY LOW RESOLUTION GC/MS

Prepared by:

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8/29/9/ Date

رو *إمداع* Date

8/29/91

Date



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Section 14.0	Specific Procedures Used to Assess Data Precision, Accuracy and Completeness
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3.0 PROJECT DESCRIPTION

Enseco is committed to providing quality environmental analytical services to both the public and private sectors. To ensure the production of scientifically sound, legally defensible data of known, documentable and verifiable quality, an extensive Quality Assurance (QA) program has been implemented within Enseco. This program relies on clearly defined objectives, well-documented procedures, a comprehensive audit system, and management support, both Corporate and Regional, for its effectiveness.

This analytical quality assurance plan addresses analyzing project samples for tetra through octa dibenzo-p-dioxins and dibenzofurans. It describes the methods and procedures that are used by Enseco-California Analytical Laboratory (CAL) to ensure that definable quality, precision, accuracy, and completeness objectives are met. Procedures and policies outlined in this document serves to supplement the quality assurance objectives delineated in "Enseco Incorporated-Quality Assurance Program Plan for Environmental Chemical Monitoring", Revision 3.4, April 1991 and is based on the <u>USEPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans</u>, QAMS-005/80.



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4.0 RESPONSIBILITIES AND AUTHORITIES

The advanced technologies services group hierarchy is shown in Figure 4-1 and a schematic of the project work flow is shown in Figure 4-2. Responsibilities are outlined as follows:

DIVISIONAL QUALITY ASSURANCE DEPARTMENTS

Members

Each Regional QA Department is managed by a QA Manager with oversite from a Regional Quality Assurance Director. The QA Manager reports directly to the Regional General Manager and the Regional QA Director. The QA Manager is supported by a QA staff within the laboratory. The QA Manager is the final authority within each laboratory on all issues dealing with data quality. He/she has the authority to require that procedures be amended to discontinued or analyses suspended or repeated. He/she can make recommendations to the Regional General Manager and the Corporate QA Director regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the Division QA Manager comes directly from the Corporate QA Director.

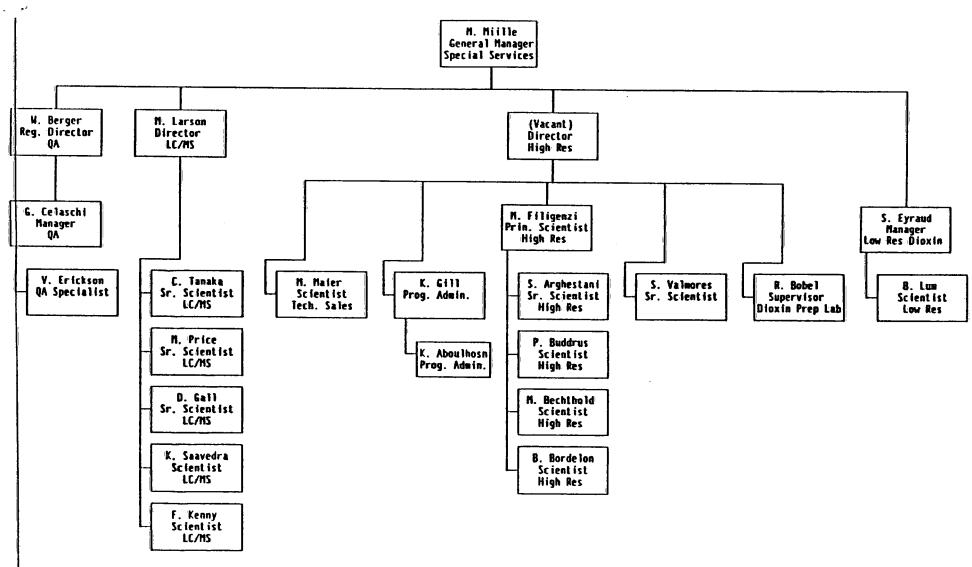
DIVISIONAL MANAGEMENT

Members

The managers, supervisors, department directors, and program administrators who direct the analytical work at each laboratory are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.



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vseco-California Analytical Laboratory
West Sacramento, California

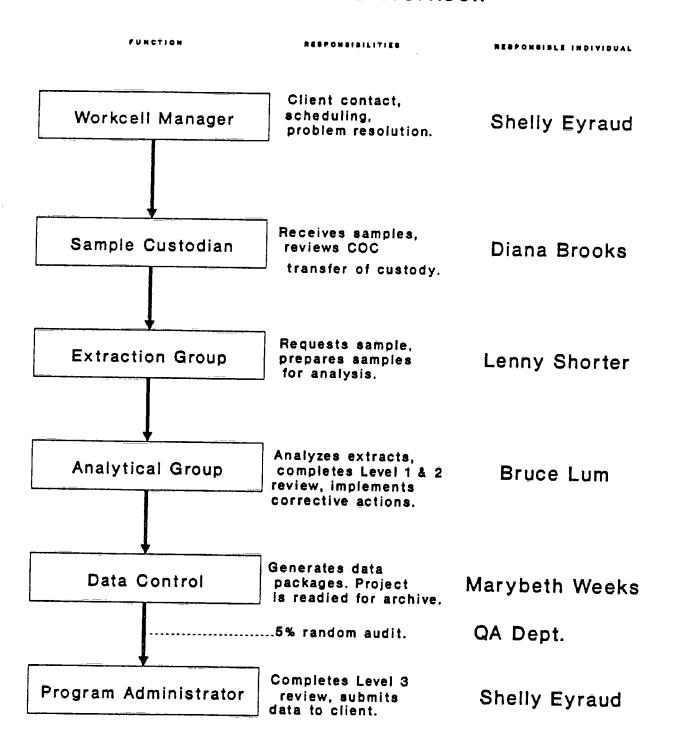
Enseco



Project Flow Figure 4.2

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Dioxin/Furans Workcell





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The managers and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgement, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the laboratory management comes directly from the President and the Regional General Manager.

DIVISIONAL PERSONNEL

Members

All laboratory personnel (including chemists, managers, etc.) involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan.

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance or rejection of data that fall outside of established QC guidelines must be approved by laboratory management and the QA department. The authority of the laboratory personnel flows from the Regional General Manager.



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5.0 QUALITY ASSURANCE OBJECTIVES

FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS,

REPRESENTATIVENESS AND COMPARABILITY

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

- DCS = Duplicate control samples, a pair of standard, control matrix that is spiked with a group of target compounds.
- RPD = Relative percent difference.
- RSD = Relative standard deviation.
- CV = Coefficient of variation.
- S = Standard deviation.
- X = A measured value.
- \overline{X} = Average. Calculated as the sum of all measured values in a population divided by the number of values in the population.
- n = Number of measurements or values in a population.

<u>Precision</u> is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with control limits established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.



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Standard deviation (S) is calculated as follows:

$$S = \sqrt{\frac{1}{\frac{1}{n-1}} \quad \frac{\frac{N}{1}}{\frac{1}{n-1}} \quad (x_1 - \overline{x})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n=2).

$$RSD = 100 (s/X)$$

or

$$CV = 100 (s/X)$$

where: RSD = relative standard deviation

CV = coefficient of variation

s = Standard deviation

X = mean



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In the case of duplicates, the $\bar{\text{RPD}}$ between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

D₁ = first sample value

D₂ = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using DCS, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing DCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

Yercent Recovery - T x 100

where: X = the observed value of measurement

T = "true" value



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Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

<u>Completeness</u> is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

valid data obtained

Completeness = total data planned x 100%

The completeness objective is 100%. Reanalysis will be performed in accordance with the procedures stated in Section 9.0, Analytical Procedures, and in Appendix I in order to meet this completeness goal.



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Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

Modified Method 8280 (Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, November 1986) will be used for the analysis of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo-furans by low resolution GC/MS. See Appendix I for the list of enhancements to Method 8280 that are part of Enseco-CAL's procedure.

Method specific accuracy and precision objectives are listed in Table 5.1.

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TABLE 5.1

ACCURACY AND PRECISION OBJECTIVES

COMPOUND	SPIKE CONCENTRATION (ng)	% RECOVERY	RPD
2,3,7,8-TCDF	10	60-140	50
1,2,3,7,8-PeCDF	10	60-140	50
1,2,3,4,7,8-HxCDF	10	60-140	50
1,2,3,4,6,7,8-HpCDF	10	60-140	50
OCDF	50	60-140	50
2,3,7,8-TCDD	10	60-140	50
1,2,3,7,8-PeCDD	10	60-140	50
1,2,3,4,7,8-HxCDD	10	60-140	50
1,2,3,4,6,7,8-HpCDD	10	60-140	50
OCDD	50	60-140	50

Units = ng/sample



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6.0 SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters;
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization;
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed;
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met.

Sample Containers and Preservatives

Enseco can assist in the sample collection process by providing consultation and assistance to client designing sampling programs. Also, Enseco can make available to the client the Enseco "Sample SafeTM", a set of sample containers that are properly cleaned and preserved for use in sample collection.



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EPA has established holding time requirements for some analyses. However, holding times for dioxins/furans can vary depending upon the method requested. EPA Method 1613A states that up to one year before extraction of aqueous and solid samples is acceptable due to the stability of dioxins and furans in the environment. This is consistent with the scientific literature which contains several references to the half-life of dioxins in soil - all of which measure the time in years. Samples will be extracted and analyzed according to a client's contract specifications.

On occasion, a sample must be reanalyzed to comply with this QA Program Plan. If this reanalysis is conducted outside of the holding time, the laboratory will be considered to have fulfilled its obligation to meet holding times if the first preparation and/or analysis was initiated within the prescribed holding time.



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7.0 SAMPLE CUSTODY

A sample is considered in custody if:

- It is in the sampler's or the transferee's actual possession;
- It is in the sampler's or the transferee's view, after being in his/her physical possession;
- It was in the sampler's or the transferee's physical possession and then he/she secured it to prevent tampering; and
- It is placed in a designated secure area.

Upon transfer of custody to Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

All samples are received by Enseco's Sample Control Group's designated sample custodian and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.



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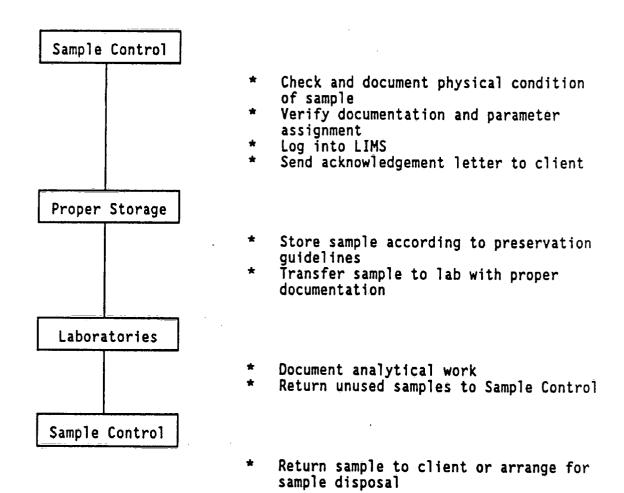
In the event that the laboratory sample custodian judges the sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), the Program Administrator (PA) will be advised immediately and the samples will not be analyzed until the PA so authorizes. The PA or designated representative will immediately contact the client. The PA and the client will make a decision as to the fate of the sample(s) in question on a case-by-case basis. The sample(s) will either be processed "as is" with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. Any problem with a sample will be noted on the chain-of-custody form.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2.



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FIGURE 7-1 ENSECO SAMPLE PROCESSING FLOW CHART



		1	1 C C O		· ····Linsaco
			STODY RECORD	Section 7.0 Revision 0	Augusts 1994 Page 21 of
SAMPLER (Signature) Phone		Date Shinned	Date Shipped		
		Airbill No		Cooler No.	
			SEND RESULTS TO:		
SHIP TO:	Enseco-Cal Lab			· 	
	2544 Industrial Blvd.		- ·		
	West Sacramento, CA 95691		Address		
	(916) 372-1393				
ATTENTION-	(710) 572-1575	;; -	Phone		
					
PROJECT	NAME		PROJECT NO.	P.O	. NO
Relinquish	ed by: (Signature)	Received by	y: (Signature)	Date	Time
Relinquish	ed by: (Signature)	Received by	y: (Signature)	Date	Time
Relinquish	ed by (Signature)	Received at	l lab by: (Signature)	Date	Time
Relinquish	ed from lab by: (Signature)	Received by	y: (Signature)	Date	Time
		ANALYSIS	REQUEST		
Sampl		Date Time	A - Lucia D		Sample Condition
Num!		Sampled	Analysis Re	·	Upon Receipt
				 	
					
	tructions Comments:				
		OF NON-AQUEO		E RETURNED TO CLI	ENT
Special Ins	tructions Comments:	OF NON-AQUEOL		E RETURNED TO CLI	ENT
	tructions Comments:			E RETURNED TO CLI	ENT



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8.0 ANALYTICAL CALIBRATION PROCEDURES AND FREQUENCY

Standard/Reagent Preparation

To ensure the highest purity possible, all primary reference standards and standard solutions used by Enseco are obtained from the National Institute of Standards and Technology, the EPA Repository or other reliable commercial sources. Dioxins/furans standards will be purchased from Cambridge Isotope Laboratories, Woburn, Massachusetts or from the US EPA, EMSL-Las Vegas (when available). All standards and standard solutions are logged into a data base that identifies the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Specific procedures are described as follows:



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Tuning

Mass calibration of the mass spectrometer is tuned prior to the analysis of standards or samples each analysis day. The compound FC43 is used to tune the instrument for greater sensitivity in the high mass range to achieve better response in the later eluting compounds.

Window Defining Mix (WDM)

The window defining mix is analyzed to verify that the switching times between the descriptors have been appropriately set and is analyzed for the following conditions:

- Before initial calibration on each instrument and on each gas chromatography column used for analysis.
- Each time a new initial calibration is performed, regardless of reason.
- Each time that adjustments or instrument maintenance activities are performed that may affect retention times.

Initial Calibration

Five calibration solutions (CC1-CC5) containing 10 unlabelled and 6 carbon labeled PCDDs/PCDFs at known concentrations are used to calibrate the instrument before sample analysis can commence. Analytes and concentrations are listed on Table 8.1. The relative ion abundance ratios (using areas to calculate the ratios) must be within the limits outlined in Table 8.2 In addition, all analytes must fall within the retention time windows as determined by the WDM and meet the mass spectrometer sensitivity criteria, i.e., the signal-to-noise ratio must be greater than 2.5 for the unlabelled PCDDs/PCDFs ions and greater than 10 the the internal standard ions. The %RSD of the RRF's for the unlabelled PCDDs/PCDFs, the surrogate, and the internal standards must not exceed 15%. Formulas used to calculate %RSD and RRF are listed in Section 14.0.



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Continuing Calibration Standard (Daily Standard)

The daily standard is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance. The RRF of the compounds in the continuing calibration standard must be within 30% of the average RRF determined from the Initial Calibration. If this criteria cannot be met, analysis is suspended, the problem investigated, corrective actions implemented, and a new 5 point calibration is performed, if needed.

Optimum Range

The optimum concentration range of this method is 0.5 - 10 ppb.

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TABLE 8.1

Concentration Calibration Solutions*
Used for Initial Calibration

	Low I	Mid-Low	Med.	Mid-high	High
2,3,7,8-TCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,7,8-PnCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,6,7,8-HxCDF	0.2	0.5	1.0	2.0	5.0
1,2,3,4,6,7,8-HpCDF	0.2	0.5	1.0	2.0	5.0
OCDF	0.40	0.50	2.0	4.0	5.0
2,3,7,8-TCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,7,8-PnCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,6,7,8-HxCDD	0.2	0.5	1.0	2.0	5.0
1,2,3,4,6,7,8-HpCDD	0.2	0.5	1.0	2.0	5.0
OCDD	0.40	0.50	2.0	4.0	5.0
13C-2,3,7,8-TCDF	0.5	0.5	0.5	0.5	0.5
13C-2,3,7,8-TCDD	0.5	0.5	0.5	0.5	0.5
13C-1,2,3,7,8-PnCDD	1.0	1.0	1.0	1.0	1.0
13C-1,2,3,6,7,8-HxCDD	1.0	1.0	1.0	1.0	1.0
13C-1,2,3,4,6,7,8-HpCDD	1.0	1.0	1.0	1.0	1.0
13C-0CDD	5.0	5.0	5.0	5.0	5.0
37C1-2,3,7,8-TCDD	0.2	0.2	0.2	0.2	0.2

^{*} Concentrations are in ng/ul.



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TABLE 8.2

Criteria for Isotopic Ratio Measurements for PCDDs and PCDFs

	<u>Analyte</u>	Selected ions	Relative Intensity
PCDFs	Tetra	304/306	0.65-0.89
	Penta	340/342	1.31-1.77
	Hexa	374/376	1.05-1.41
	Hepta	408/410	0.88-1.18
	Octa	442/444	0.75-1.01
PCDDs	Tetra	320/322	0.65-0.89
	Penta	356/358	1.31-1.77
	Hexa	390/392	1.05-1.41
	Hepta	424/426	0.88-1.18
	Octa	458/460	0.75-1.01
Interna	l Standards		
	13C-TCDF	316/318	0.65-0.89
	13C-2,3,7,8-TCDD	332/334	0.65-0.89
	13C-PnCDD	368/370	1.31-1.77
	13C-HxCDD	402/404	1.05-1.41
	13C-HpCDD	436/438	0.88-1.18
	13C-OCDD	470/472	0.75-1.01
Recover	y Standard		
	13C-1,2,3,4-TCDD	332/334	0.65-0.89



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9.0 ANALYTICAL PROCEDURES

The methods used are those specified by the US EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including chlorinated dioxins and furans.
- "Test Methods for Evaluating Solid Waste" (SW-846), 2nd Edition (revised), Update I (1984), Update II (1985), 3rd Edition (1986), Update I (1989), Office of Solid Waste and Emergency Response, US EPA.

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. The method selected will detect and quantify 2,3,7,8-tetrachlorinated dibenzo-p-dioxin (2,3,7,8-TCDD), 2,3,7,8-tetrachlorinated dibenzofuran (2,3,7,8-TCDF), and the 2,3,7,8-substituted penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs). Any deviations from published methodology are documented and explained in Appendix I. Samples that initially do not meet quality assurance objectives will be reanalyzed once to verify observed anomalies. Additional reanalysis can be performed at the request of the client for a minimal fee. Specific analytical procedures are as follows:



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Internal Standards

To quantitate and measure recovery of the analytes, labeled internal standards will be added to all pre-extraction samples, QC samples, blanks, extraction, and to calibration solutions. Calculated internal standard recoveries must be >=40% or the signal-to-noise ratio must be at least 10:1 before data is considered acceptable. Otherwise, samples will be reextracted and reanalyzed at a smaller sample volume. Associated analytes and internal standards are listed in Appendix I.

Surrogate

The surrogate 37C1-2,3,7,8-TCDD will be added to all pre-cleanup blanks, samples, QC samples, and to calibration solutions. The calculated % recovery is used to verify the recovery of unlabelled PCDDs/PCDFs and to monitor the efficiency of the cleanup procedures.

Recovery Standard

To measure the % recovery of the labeled internal standards, the recovery standard 13C-1,2,3,4-TCDD will be added to all blank, sample, and quality control sample extracts just prior to GC/MS analysis.

Additional Quality Control

In addition, the following quality control samples will be analyzed:

- Method blanks
- Matrix spike/matrix spike duplicate sample
- Duplicate control sample (DCS)

Definitions, frequency, and corrective action are discussed in Section 11.0, Internal QC Checks and Frequency. Acceptability criteria are listed in Table 5.1, Accuracy and Precision Objectives.



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<u>Calculations</u>

A summary of calculations used to determine PCDF/PCDD recoveries and concentrations are listed in Section 14.0, Specific Procedures Used to Assess Data Precision, Accuracy, and Completeness.

<u>Identification</u>

For a gas chromatographic peak to be unambiguously identified as a PCDD/PCDF, it must met the following criteria:

- The absolute retention times must be within method acceptance limits.
- All of the specified ions listed in Appendix I for each PCDD/PCDF must be present in the ion current profile. The ion current response for the two quantitation ions and the M-[COCI]+ ions for the analytes must maximize simultaneously.
- The integrated ion current for each analyte ion listed in Appendix I must be at least 2.5 times background noise with no detector saturation.
- Ion abundance ratios must meet the criteria listed in Table 8.2.

<u>Confirmation</u>

Because specificity for all of the isomers cannot be achieved on a 60 M DB-5 chromatographic column, a second column (SP-2331) will be used to confirm the presence of any 2,3,7,8-substituted PCDDs/PCDFs detected. Samples are first analyzed on a GC/MS fitted with a 60 M DB-5 chromatographic column. If any 2,3,7,8-substituted tetra-, penta-, or hexa- PCDDs/PCDFs are detected, the sample extracts will be reanalyzed using a 60 M SP-2331 chromatographic column. If data resulting from SP-2331 column does not confirm results from the DB-5 column, only data calculated from the SP-2331 column will be reported. All data will be corrected following proper error correction protocol.



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10.0 DATA REDUCTION, VALIDATION, AND REPORTING

All analytical data generated within Enseco laboratories are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review, as described below (also see Figure 10-1).

Level 1 Review

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs.

Data will be reduced by an analyst in one of the following ways:

- Manually computing results directly on the data sheet, chromatogram, or on calculation pages attached to the raw data;
- Inputting raw data for computer processing.

If data are manually reduced by an analyst, all steps in the computation will be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and RFs, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than).



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Finally, each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample identification is correct;
- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met;
- · Congener identification is correct; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, Out-of-Control forms [if required] are complete; holding times are documented, etc.).

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.



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Level 2 Review

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct:
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; Out-of-Control forms [if required] are complete; holding times are documented, etc.).
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.



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Level 3 Review

Before the report is released to the client, the Program Administrator who is responsible for interfacing directly with the client reviews the report to ensure that the date meet the overall objectives of the client, as understood by the Program Administrator. This review is labeled Level 3 review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgement of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. Errors that are found are documented and transmitted to the appropriate supervisors and managers. The cause of the errors is then addressed with additional training or clarification of procedures to that quality data is generated from the lab. The process continues until no errors are found or until the data package has been reviewed in its entirety.

<u>Detection Limits</u>

The detection limit will be calculated for the 10 congeners when no unlabelled PCDFs/PCDDs are detected in the samples. The process is discussed in Appendix I. Calculations are listed in Section 14.0.



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Reporting

Results will be reported to the second decimal place in units of ng/l (ppt) for aqueous samples, and ng/g (ppb) for solid samples on a wet weight basis.

An example of a dioxin/furan data validation scheme is shown as Figure 10-1.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

- General Discussion: Description of samples types, tests performed, any problems encountered and general comments are given.
- Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.
- OC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

Results of any matrix spikes, duplicates, matrix spike duplicates or other project-specific QC are also reported.

- · Methodology: Reference for analytical methodology used is cited.
- Raw Data: Including calibration data, window defining mix data are included in CLP-type deliverables.



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Data Validation

Data validation involves reviewing sample results, quality control, and procedures for compliance with the data quality objectives outlined in this document or with client specified objectives. The scheme used for validation of data is shown in Figure 10.1.

Section 10.0 Figure 10-1 Revision 0 August 1991 **Data Validation Scheme** Page 36 of 53 Sample Analysis Chemist No Reanalyze Results Reviews if Necessary Acceptable Data Ycs Data No Reanalyze Results Review Acceptable if Necessary Specialist Ycs Results Client Manager Acceptable Quality 5% Audits Assurance Office Hardcopy Report to Client



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11.0 INTERNAL OC CHECKS

Laboratory Performance QC samples will be added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration so that accuracy and precision can be determined and control charts can be prepared. Measures taken to control analytical data quality include use of specific acceptance criteria for instrument calibration, laboratory control samples, duplicate analyses, blank samples, and spiked samples.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks; and
- The generation of daily calibration data.

Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS consists of a standard, control matrix that is spiked with the internal standards and the surrogate representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.



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Accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference [RPD] between each analyte in the DCS pair) data are compared to control limits that have been established for each of the analytes contained in the DCS. Initially, control limits for analytes spiked into the DCS are taken directly from the CLP program. If CLP limits are not available, Enseco historical data are used to set the control limits. As sufficient laboratory data become available, the control limits are redefined based upon the most recent nine months of DCS data. Control limits for accuracy for each analyte are based on the historical average recovery (mean of the average recoveries of the DCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte are based on the historical RPD and range from zero (no difference between DCS results) to the average RPD plus three standard deviation units.

Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. The procedure used to evaluate data from control samples is given in Figure 11-1. The protocols include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

Method Blank

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

Laboratory Performance QC Control Sample Evaluation August 1991 Page 39 of 53 **Poratory Control** mple Generated DOS/SCS Data Analyzed **Acceptable** Yes Report data with all associated No samples Validate instrument Refer problem operational settings. to Supervisor sensitivity & linearity Confer with Problem Identified Can all samples is problem related Sample Prep be reextracted? only to DCS/SCS Group Yes Yes Correct & reanalyze No Report data with all associated Data samples **Acceptable** (w/explanation) Reextract & **Document on** Reanalyze DCS/SCS Form Yes Report data with Report data with all associated Report data with all associated samples all associated samples (w/explanation) samples



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As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

A method blank is prepared and analyzed with every analytical lot or for every 20 samples, whichever is more frequent. Any analyte detected in the blank must be below the reporting limit, otherwise samples are reextracted and reanalyze.

Matrix-Specific QC

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, Enseco encourages its clients to include Matrix-Specific QC that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.



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A Matrix Spike Duplicate (MSD) is an environmental sample that is divided into two separate aliquots, each of which is spiked with known concentrations of the analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

Surrogate Compounds

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. A labeled surrogate will be added to all extracted, pre-cleanup blanks, samples, QC samples, and to calibration solutions to monitor the efficiency of the cleanup procedure.

Field Blank

Field blanks are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. Solvents such as trichloroethylene are the medium of choice for field blanks when sampling for dioxins and furans; however, if solvents are also parameters of interest, no field blank will be collected. One example of a field blank is an equipment blank. Another type of field blank is a trip blank. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample container, remains unopened in the field, and then is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks. The purpose of the trip blank is to assess the impact of field and shipping condition on the samples.



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12.0 PERFORMANCE AND SYSTEMS AUDIT

Enseco laboratories participate in a variety of federal and state certification programs, (including the US EPA CLP), that subject each of the laboratories to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the US EPA, US Army, US Navy, and over 4 states. The most current list of Enseco certification is available upon request.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

Ciba-Geigy, Inc. performed both a system and a performance audit for this project. Two 2,3,7,8-TCDD performance evaluation samples were supplied by EPA Region I and submitted to the laboratory for concurrent analysis with samples. Diana Baldi and Frank Saksa of Ciba-Geigy conducted a systems audit while samples were being processed by the laboratory.

A summary of the types and frequency of systems and performance audits is summarized in Table 12.1.



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TABLE 12.1 SUMMARY OF PERFORMANCE AND SYSTEM AUDITS

PRCGRAM	NAME	TYPE (P OR S)	FREQUENCY
Drinking water	WS	Performance	Semi-Annual
Waste water	WP	Performance	Semi-Annual
EPA-CLP	QB	Performance	Quarterly*
U.Ś. Navy	PE	Performance	Every 18 mos.*
U.S. Army	PE	Performance	Every 18 mos.*
Calif. Dept. Food			
and Agriculture	PE	Performance	Quarterly
NPDES	DMR-QA	Performance	Annually
EPA-CLP		System	Annually*
Calif. ELAP		System	Biannually
U.S. Navy		System	Annually
Utah		System	Annually
U.S. Army		System	Every 18 mos.*
Divisional QA		System	Quarterly
			(approx.)
ENSECO Corporate		System	Annually
Ciba-Geigy, Inc.		System	Random

Clients may request performance of specific performance and systems audits as a requirement of contract award.

^{*} Contract award required.



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13.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and schedules.

All aspects of routine and non-routine instrument maintenance are recorded in logbooks, and a log book is dedicated to each instrument.

Balances are calibrated daily or as used with Class S or Class S traceable weights at specific weights of use with the results entered in a logbook kept near the balance.

Ovens and refrigerators are fitted with uniquely marked thermometers and monitored daily. Limits for refrigerators are 2°C to 6°C. If a temperature falls outside these limits, the appropriate laboratory manager is alerted and corrective action is taken. The readings are entered in a logbook kept near the thermometer. Annually the thermometer is calibrated vs. an NIST traceable thermometer.



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14.0 SPECIFIC PROCEDURES USED TO ASSESS DATA PRECISION ACCURACY. AND COMPLETENESS

Calculations for accuracy (% R) and precision (RPD, RSD) were discussed and defined in Section 5.0 Data Quality Objectives. Formulas used to calculate relative response factors (RRF), concentration, and detection limits are as follows:

Definitions

Cn = concentration of native PCDD/PCDF found in the sample

Cis = concentration of internal standards

W = weight (g) of sample extracted

V = volume (mL) of sample extracted

Qis = quantity (ng) of internal standard added to sample before extraction

Qrs = quantity (ng) of recovery standard added to sample extract

Qs = quantity (ng) of surrogate added to sample before extraction

An = integrated ion abundance of the quantitation ion of the isomer of interest (Table 1).

Ais = integrated ion abundance of the quantitation ion of the appropriate internal standard (Table 1 and 2).

RRFn is the response factor of the quantitation ion of the isomer of interest relative to that of the appropriate internal standard.

RRFs is the response factor of the quantitation ion of the surrogate relative to that of the appropriate internal standard.

RRFis is the response factor of the internal standard relative to that of the appropriate recovery standard.



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Relative Response Factors

<u>Concentration</u>

The concentrations of the various isomers of each congener shall be calculated using the RRF determined for that particular isomer from the initial calibration.

Detection Limits

For samples in which no unlabelled PCDFs/PCDDs are detected, calculate the detection limit for each of the 10 congeners. The DL is the concentration of a given PCDF/PCDD congener that would produce a signal with peak height of 2.5 times the background signal level, or when no interference is present, the equivalent concentration that the peak would represent if it were a PCDF/PCDD congener. The data must be carefully examined to determine which DL calculation will be used. Follow the rules below.

When only electronic noise is present, or if only one chemical peak is present at either the primary or secondary ion, use the following calculation:

When chemical peaks are present at both the primary and secondary ions that do not meet ratio criteria use formula 2 below:

$$MPC = \frac{Hx \times Qis}{His \times RRFn \times (W \text{ or } V)}$$



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15.0 CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- · Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department. Corrective action documentation is routinely reviewed by the of QA.



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16.0 OA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Regional QA Directors submit extensive monthly reports to the Corporate QA Director and the Regional General Manager. These reports include:

- The results of internal systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the 5% QA data audits conducted.

The Corporate Director of QA submits regularly reports on the status of the QA program to the President and Regional General Manager. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.



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APPENDIX I PCDDS/PCDFS

Enseco-Cal Lab performs United States Environmental Protection Agency (US EPA) Method 8280 for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) with some minor modifications to the published method. Since the method was first published in the Federal Register in September 1986, this method has undergone several updates and changes by EPA to keep pace with technology and method improvements during that time period.

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 M DB-5 column. In order to determine the concentrations of the individual 2,3,7,8-substituted isomers, the sample extract shall be reanalyzed on a 60 M SP-2331 GC column. The chromatographic resolution is evaluated using a commercially available column performance mixture containing the TCDD isomers that elute most closely with 2,3,7,8-TCDD.

GENERAL DIFFERENCES

<u>Calculation of Detection Limits</u>

The sensitivity of this method is dependent upon the level of interferences within the sample matrix. All PCDD and PCDF analyses performed for EPA since 1982 has used a technique for calculating the detection limit for each of the chlorination levels and each congener by using the noise level present in the elution window and the height of the chromatographic peak of the internal standard. Both the signal to noise and peak height are determined by the data system of the GC/MS. The result of the calculation is a detection limit that is specific to the homologous series and sample. We are not aware of any laboratories in the dioxin field that use or have used the MDL study referenced in the original 8280 method.



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Internal Standards

Due to the increased availability of the 13 C-labeled PCDD and PCDF internal standards, Enseco-Cal Lab currently uses six instead of only the two internal standards specified and one recommended in the method. This clearly improves the quality of the data.

Internal Standards and Corresponding Analytes

13C-TCDD	13C-TCDF	13c-PnCDD	13c-HxCDD	13c-HpCDD	13C-OCDD
TCDD	TCDF	PnCDF	HxCDF	HDCDF	OCDF
37C1-TCDD		PnCDD	HxCDD	HpCDD	- OCDD

Reporting Format

The reporting forms at the end of Method 8280 have been revised a number of times to improve the initial version. Enseco-Cal Lab results, while not reported on the forms in Method 8280, are in a "CLP-like" format.

Initial Calibration

The initial calibration is performed with single injections of a five point curve.

Acceptance of Internal Standard Recoveries

Method 8280 specifies internal standard recoveries must be 40% or greater. If recoveries do fall below 40%, then the signal-to-noise ratio is calculated. The recoveries of the internal standards are judged acceptable if the signal-to-noise ratio is greater than 10:1.

Key Ions Used in the Analysis

Below are the ions used in the determination of PCDDs and PCDFs, they will differ from those listed in Method 8280. The quality of data will not be effected as the ions are consistently used in both the analytical standards and samples.



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Ions Specified for Selected Ion Monitoring for PCDDs and PCDFs

<u>Analyte</u>	Quantitation <u>ion</u>	Confirmation <u>ion</u>	M-[COC1] +
PCDFs			
Tetra Penta Hexa Hepta Octa	306 340 376 410 444	304 342 374 408 442	243 277 311 345 379
PCDDs			
Tetra Penta Hexa Hepta Octa	322 358 392 426 460	320 356 390 424 458	259/257 293 327 361 395
Internal Standards			
13C-TCDF 13C-2378-TCDE 13C-PnCDD 13C-HxCDD 13C-HpCDD 13C-OCDD	318 334 370 404 438 472	316 332 368 402 436 470	

PCDD/PCDF isomers in the window defining mix for a 60 M DB-5 (or equivalent) column.

<u>Homologue</u>	First <u>Eluted</u>	Last <u>Eluted</u>
TCDD TCDF PeCDD PeCDF HxCDD HxCDF HpCDD	1368- 1368- 12478 13468- 124679- 123468- 1234679-	1238- 1289- 12389- 12389- 123467- 123489- 1234678-
HPCDF	1234678-	1234789-



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SPECIFIC DIFFERENCES

Analytical

Section 4.3.2 - 8280: 30 M DB-5

Cal: 60 M DB-5

Section 10.3 - 8280: Recommended that the GC/MS run be

divided into five selected ion

monitoring sections.

Cal: GC/MS run is divided into three selected

ion monitoring sections.

Section 11.1 - 8280: Response factor of the quantitation ion

(m/z 334) of the internal standard, 13c-

2,3,7,8-TCDD.

Cal: Response factor of the quantitation ion

of the compound of interest relative to the appropriate $^{13}\text{C}_{12}\text{-labelled}$ internal

standard.

Sample Preparation

Section 9.2.5 - 8280: Soil extraction with 20 mL methanol in

and 80 mL petroleum ether.

Cal: 20 mL methanol and 150 mL hexane.

Acceptable internal standard recoveries have been demonstrated using hexane as

an extraction solvent.

Section 9.2.5.1 - 8280: Kuderna-Danish concentration.

Cal: Concentration performed by rotary

evaporation.

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Section 9.3 - 9.6 8280: Regents used 20% KOH, 5% NaCl

Cal: Regents used 10N NaOH, DI H2O

The acid/base wash is performed on an optional basis. A silica/alumina column cleanup is the first column cleanup performed and follows (Section 11.3) from SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. The second column is the carbon column cleanup. This cleanup is also from SOW 9/86, Rev. 8/87, Section 11.4.3 - 11.4.5 with omitting 5% benzene as the only modification.

Section 9.7 8280: Gravity column with 4 g of Woelm super 1 neutral alumina.

Cal: Two columns in series. The first consisting of 1 cm Na₂SO₄, silica gel, 4 g 44% H₂SO₄ (silica gel, 1 g silica gel, 2 g 33% 1M NaOH/silica gel, 1 g silica gel and glasswool. The second consisting of 1 cm Na₂SO₄, 6g acid alumina and glasswool. The above column packing materials can be found in Section 11.3, SOW 9/86, Rev. 8/87, Form IFB Series WA 86-K357. This cleanup is mandatory.



SAMPLING AND ANALYSIS OF LANDFILL GAS FOR POTENTIALLY CORROSIVE ORGANIC AND INORGANIC CONTAMINANTS



SAMPLING AND ANALYSIS OF LANDFILL GAS FOR POTENTIALLY CORROSIVE ORGANIC CONTAMINANTS

General

Core Laboratories initially became involved in the analysis of landfill gas in March, 1987. We were contracted to investigate the cause of corrosion occuring in a combustion engine using landfill gas as a fuel. Sulfur compounds such as hydrogen sulfide are known to be corrosive and are commonly found in natural gas but are not commonly found in landfill gas. Halogenated hydrocarbons have been found in landfill gas in varying concentrations and it has been theorized that these compounds when combusted and combined with water vapor in the exhaust gases may form highly corrosive by-products.

Methodology

There are few published methods for the analysis of gaseous samples for volatile organic compounds and these are more applicable to industrial emissions (CFR 40 - Pt. 60, App. A, Method 18) and ambient air monitoring for health related exposure (NIOSH - Manual Analytical Methods, Third Edition). Gas chromatography coupled with mass spectroscopy (GCMS) is the most effective methodology when analyzing samples for several volatile organic compounds. EPA method 624 is a recognized GCMS method for analyzing water and soil samples for a specific list of volatile compounds. Personnel at Core Laboratories initially used a modification of this procedure when analyzing gaseous samples. Problems with instrument calibration techniques and questions concerning the stability of containerized gaseous samples led Core Laboratories to purchase a thermal desorption unit in July of 1987. A more accurate and reliable calibration method was developed and by using an adsorbing tube sampling technique sample integrity was assured.

In April, 1987 Core laboratories personnel developed a different method for analyzing landfill gas that would be easier and less expensive to perform than traditional GCMS methods. This method involves first passing a sample of gas through a glass tube containing granular activated carbon and then combusting the carbon at high temperatures and measuring the quantity of certain halide ions produced (Cl⁻, Br⁻, I⁻). This quantity represents the total amount of certain halogenated hydrocarbons in the gas and is expressed in ug/l as Cl⁻ at STP (0°C, 760mm Hg). This method closely approximates the processes taking place in a combustion engine and provides a reliable quantification of corrosion potential in the fuel gas.



ANALYSIS OPTIONS

Core Laboratories offers complete characterization of landfill gas quality through several different methods of analysis. The following is a list of analysis options and their application towards characterization.

1. Volatile Organic Compounds* (Molecular weight range - 40 to 260)

A thermal desorption technique coupled with GCMS (TD/GCMS) is utilized for measuring all detectable quantities of organic compounds in the molecular weight range of 40 to 260. An adsorption tube sampling method eliminates questions concerning sample stability associated with various sample container types. All detected compounds are reported as ug/l at STP (0°C, 760mm Hg).

2. Total Organic Halide*

This analysis measures the total amount of certain halogenated (containing chloride, bromide, or iodide) organic compounds present in the gas and expresses this quantity in ug/l as Cl at STP. Again an adsorption tube sampling method is utilized assuring sample integrity. The values generated by this method can be used to check analysis for volatile organic compounds. It should be noted that the quantities of chloride, bromide and iodide are all expressed in one value as chloride. Due to the limitations of this methodology the quantity of fluoride is not included.

3. Sulfur Compounds

All sulfur containing compounds are measured in gas samples using a gas chromatograph equipped with a sulfur specific flame photometric detector and a Chromosil 330 column. Concentrations down to 0.001 mole % can be detected in this analysis. Teflon lined cylinders are recommended for sampling.

4. GPA Fuel Gas Analysis

Standard methods documented by the Gas Producers Association are used to measure major constituents in any type of fuel gas. Lower Heating Value (LHV) is calculated from the gas make-up and reported in BTU per cubic foot of dry gas at 60°F and 762 mm Hg. A cylinder or tedlar bag is suitable for sampling when performing this analysis.

* Sampling kit necessary to perform sampling of landfill gas to analyze for volatile organic compounds or total organic halide.





ANALYSIS PRICING

Analysis Type	Cost per Sample
Volatile Organic Compounds	\$375.00
Total Organic Halide	\$95.00
Sulfur Compounds	\$175.00
GPA Fuel Gas Analysis	\$110.00



SAMPLING GAS FOR VOLATILE ORGANIC COMPOUNDS

While developing methodology for analyzing fuel gases for total organic halide and volatile organics several questions were raised pertaining to the stability of containerized gaseous samples. It became apparent that volatile organic compounds in containerized gas samples are not stable over time. The sample container types studied include stainless steel cylinders, teflon-lined stainless steel cylinders and Tedlar bags.

As a result of concerns about the integrity of containerized gas samples Core Laboratories personnel sought a new more reliable sampling technique. The technique developed involves taking a sample of gas of known volume and passing it through a glass tube containing an adsorbent. This procedure is performed in the field at the sampling source using a sampling kit. The column containing the adsorbent is then sealed in a glass vial and returned to the lab for analysis.

SAMPLING OPTIONS USING ABSORBING TUBE SAMPLING TECHNIQUE

1. Purchase of Core Laboratories Sampling Kit (\$575.00)

Select this option when ongoing landfill gas quality monitoring is anticipated. Client would be responsible for performing sampling.

2. Lease of Core Laboratories Sampling Kit (\$100.00/week)

Select this option when a one time or initial landfill gas characterization is desired. Client would be responsible for performing sampling.

3. Both Sampling and Analysis Performed by Core Laboratories Personnel

Select this option when trained personnel for sampling are required or when ongoing landfill gas quality monitoring is anticipated and initial training on the use of sampling kit is desired. Trained Core Laboratories personnel would travel to sampling site to perform sampling. Sample training could be provided to on site personnel for future sampling.

\$100.00/week Sample Kit \$25.00/hr - travel time \$50.00/hr - sampling time + Expenses (travel, lodging etc.)



Core Laboratories Gas Sampling Kit

Co	mponent Name	Oty.	Replacement Cost Ea.	Total
1.	Glass Syringe 100cc with male Luer Lock tip	1	\$225.00	\$225.00
2.	S.S. syringe valve	1	\$40.00	\$40.00
3.	TOX Sample Tube Holder	1	\$95.00	\$95.00
	-GAC Column Housing	1	\$50.00	
	-Viton O-rings	6	\$1.50	
	-S.S. swagelok union (B.T.)	1	\$8.00	
	-female Luer tip	1	\$28.00	
4.	Volatile Organics Sample Tube Holder	1	\$40.00	\$40.00
	-S.S. Swagelock Union	1	\$8.00	
	-female Luer tip	1	\$28.00	
	-Vespel ferrule	1	\$4.00	
5.	Sampling Train	1	\$125.00	\$125.00
	-oneway S.S. check valve	2	\$34.00	
	-S.S. union T-fitting	1	\$20.00	
	-female Luer tip	1	\$28.00	
	-S.S. male pipe fitting	1	\$9.00	
6.	Carrying case	1	\$100.00	\$100.00
7.	Complete Sampling Kit	1	\$625.00	\$625.00

(complete kit includes all of above and complete instructions)



July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled: 06/25/95 Date Received: 06/27/95

GPA FUEL GAS ANALYSIS		
COMPOUND	MOLE PERCENT	CONTRIBUTION TO LHV (BTU)
OXYGEN	2.86	
HYDROGĒN	0.00	*
NITROGEN	12.0	
CARBON DIOXIDE	38.7	ਦ ਦ ਲਾ
METHANE	48.7	443
CARBON MONOXIDE	0.00	===
ETHYLENE	0.00	
ETHANE	0.00	
PROPANE	0.00	
ISO-BUTANE	0.00	
N-BUTANE	0.00	
HEXANES	0.00	
HEPTANES PLUS	0.00	
PERCENT RECOVERY	102.3	443

LOWER HEATING VALUE (calculated) = 443 BTU per cubic foot of dry gas at 60°F and 762 mm Hg



July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled: 06/25/95 Date Received: 06/27/95

ANALYSIS FOR SULFUR COMPOUNDS BY GAS CHROMATOGRAPHY

COMPOUND	MOLE PERCENT
HYDROGEN SULFIDE	0.005
CARBON DISULFIDE	ND(0.001)
CARBONYL SULFIDE	ND(0.001)
METHYL MERCAPTAN	ND(0.001)
ETHYL MERCAPTAN	ND(0.001)
n-PROPYL MERCAPTAN	ND(0.001)
iso-PROPYL MERCAPTAN	ND(0.001)
n-BUTYL MERCAPTAN	ND(0.001)
sec-BUTYL MERCAPTAN	ND(0.001)
tert-BUTYL MERCAPTAN	ND(0.001)
iso-BUTYL MERCAPTAN	ND(0.001)
n-AMYL MERCAPTAN	ND(0.001)
tert-AMYL MERCAPŢAÑ	ND(0.001)
tert-AMYL MERCAPTAN	ND(0.001)
n-HEXYL MERCAPTAN	ND(0.001)
n-HEPTYL MERCAPTAN	ND(0.001)

ND = NOT DETECTED AT LEVEL SHOWN IN PARENTHESIS



July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled:
Date Received:

06/25/95 06/27/95

TOTAL ORGANIC HALIDE 460 ug/l Cl= (measured)

* All values reported at STP (0°C, 760 mm Hg)

 $^{
m 1}{
m Due}$ to limitations in the methodology this does not include fluoride.





CORE LABORATORIES

Example of Gas Analysis Report

July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled: 06/25/95 Date Received: 06/27/95

ANALYSIS FOR VOLATILE ORGANIC COMPOUNDS (TD-GC/MS)

COMPOUND	<u>ug/l</u> *	ug/l Cl- <u>as Cl-</u>	ug/l F- <u>as F-</u>	ug/l TOTAL ORGANIC HALIDE as Cl- (TOH/CL)
Chlorobenzene	23.5	7.4		7.4
Chloroethane	6.1	3.4		3.4
Chloroform	21.0	18.7		18.7
Cis-1,2-Dichloroethene	94.7	69.3		69.3
Dichlorodifluoromethane	18.4	10.8	5.8	21.6
1,1-Dichloroethane	130	93.1		93.1
1,1-Dichloroethene	10.3	7.5		7.5
Methylene Chloride	45.0	37.6		37.6
Tetrachloroethene	67.1	57.4		57.4
Toluene	247			
Trichloroethene	150	121		121
Vinyl Chloride	23.5	13.3		13.3
TOTAL	837	440	3	451

^{*} All values reported at STP (760mm Hg, 0°C)



CORE LABORATORIES

Example of Gas Analysis Report

July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled: 06/25/95 Date Received: 06/27/95

ANALYSIS FOR VOLATILE ORGANIC COMPOUNDS (TD-GC/MS)

COMPOUND	PPMV*	PPMV C1- as C1-	PPMV F- as F-	PPMV TOTAL ORGANIC HALIDE as C1- (TOH/CL)
Chlorobenzene	4.7	4.7		4.7
Chloroethane	2.1	2.1		2.1
Chloroform	3.9	11.8		11.8
Cis-1,2-Dichloroethene	21.9	43.8		43.8
Dichlorodifluoromethane	3.4	6.8	6.8	13.6
1,1-Dichloroethane	29.4	58.9		58.9
1,2-Dichloroethylene	2.4	4.8		4.8
Methylene Chloride	11.9	23.7		23.7
Tetrachloroethene	9.1	36.3		36.3
Toluene	60.1			50.5
Trichloroethene	25.6	76.7		76.7
Vinyl Chloride	8.4	8.4		8.4
TOTAL	183	278	7	285

^{*} All values reported at STP (760mm Hg, 0°C)



July 4, 1995

SOME COMPANY INC.

Core Lab Sample I.D.: 950059-1

Client Sample I.D.: Well #9 - Shabougamou County Landfill

Alberta, Canada

Date Sampled: 06/25/95
Date Received: 06/27/95

VOLATILE ORGANIC COMPOUNDS IDENTIFIED BUT NOT OUANTIFIED

Cyclohexane

1,3-Dimethyl cyclohexane

1,1-Dimethyl cyclopentane

trans-1,3-Dimethyl cyclopentane

Hexane

Heptane

Methyl cyclohexane

Methyl cyclopentane

2-Methyl hexane

3-Methyl hexane

Octane analyse, opinious or interpretations contained in the port aerhand opon observations and a decrease the first transfer of the contract tran



VOLATILE ORGANIC COMPOUNDS

COMPOUND	MOLECULAR WEIGHT	PĒRCĒNT CHLORIDE	PERCENT BROMIDE	PERCENT FLUORIDE
Acetone	58.08			
Acrolein	56.06			
Acrylonitrile	53.06			
Allyl chloride	76.53	46.33		
Benzene	78.11			تته شد مله منه وند
Bromodichloromethane	163.83	43.28	48.77	==== =
Bromoform	252.77		94.85	
Bromomethane	94.95		84.17	
Bromotrifluoromethane (Freon 13B1) 148.92		53.65	38.28
2-Butanone	72.10			
Carbon disulfide	76.14			
Carbon tetrachloride	153.84	92.19		
Carbon tetrafluoride (Freon 14)	88.01		·	86.35
Chlorobenzene	112.56	31.50		
Chlorodifluoromethane (Freon 22)	86.47	41.00		43.94
Chloroethane	64.52	54.96	÷	
2-Chloroethylvinyl ether	106.55	33.28	~~~~	
Chloroform	119.39	89.10		
Chloromethane	50.49	70.22		
Chlorotrifluoromethane (Freon 13)	104.46	33.94		54.57
Dibromochloromethane	208.28	17.02	76.73	
1,2-Dibromo-3-chloropropane	236.36	15.00	67.62	
Dibromomethane	173.86		91.93	



VOLATILE ORGANIC COMPOUNDS

COMPOUND	MOLECULAR WEIGHT	PERCENT CHLORIDE	PERCENT BROMIDE	PERCENT FLUORIDE
1,2-Dichlorobenzene	147.01	48.24		
1,3-Dichlorobenzene	147.01	48.24		
1,4-Dichlorobenzene	147.01	48.24		
1,4-Dichloro-2-butene	123.99	57.19		
Dichlorodifluoromethane (Freon 12	2) 120.92	58.65		31.42
1,1-Dichloroethane	98.97	71.65		
1,2-Dichloroethane	98.96	71.66		~
1,1-Dichloroethene	96.95	73.14		
cis-1,2-Dichloroethene	96.95	73.14		
1,2-Dichloroethylene	96.95	73.14		
Dichlorofluoromethane (Freon 21)	102.92	68.89		18.46
1,2-Dichloropropane	112.99	62.76		
cis-1,3-Dichloropropane	112.99	62.76		~~~=
trans-1,3-Dichloropropane	112.99	62.76		
trans-1,3-Dichloropropene	110.98	63.90		
1,2-Dichlorotetrafluoromethane (Freon 114)	170.92	41.48		44.47
Diethyl ether	74.12			
p-dioxane	88.10			
Ethyl benzene	106.16			
Fluorotrichloromethane (Freon 11)	137.37	77.42		13.83
Ethyl-methacrylate	114.15			
2-Hexanone	100.16			



VOLATILE ORGANIC COMPOUNDS

COMPOUND	MOLECULAR <u>WEIGHT</u>	PERCENT CHLORIDE	PERCENT BROMIDE	
Hexafluoroethane (Freon 116)	138.01	====		82.60
Iodomethane	141.95	(% Iodide	e = 89.41)
Methacrylonitrile	67.09	42===		
Methylene Chloride	84.94	83.49		
Methyl ethyl ketone	72.10	÷=		
Methyl-methacrylate	86.09			
4-Methyl-2-pentanone	100.16			
Pentachloroethane	202.31	87.63		
Styrene	104.14			
1,1,1,2-Tetrachloroethane	167.86	84.49		
1,1,2,2-Tetrachloroethane	167.86	84.49		حد حدّ مد خد مد
Tetrachloroethene	165.85	85.52		
Toluene	92.13			
1,1,1-Trichloroethane	133.42	79.72		
1,1,2-Trichlorethane	133.42	79.73		
Trichloroethene	131.40	80.95		
1,2,3-Trichloropropane	147.43	72.14		
Trifluoromethane (Freon 23)	70.01			81.42
1,1,2-Trichloro-1,2,2-trifluoro-ethane	187.38	56.76		30.42
Vinyl acetate	86.09			
Vinyl Chloride	62.50	56.73		
O-Xylene	106.16			
M+P-Xylene	106.16			



CONVERSION CALCULATIONS FOR GAS ANALYSIS

CONVERSION OF SAMPLE VOLUME (AT TIME OF SAMPLING) TO SAMPLE VOLUME AT REFERENCE CONDITIONS (STP = 0°C and 760 mm Hq)

$$V_R = V X \frac{P}{760} X \frac{273}{T + 273}$$

 V_R = Sample volume at STP (0°C and 760mm Hg)

V = Sample volume at time of sampling

P = Barometric pressure at time of sampling (mm Hg). Barometric pressure corrected for altitude is uncorrected assuming a one inch drop in Hg per 1000 feet of altitude.

T = Temperature of sample at time of sampling (°C)

CONVERSION OF CONCENTRATION IN PPMV @STP TO CONCENTRATION IN ug/L @STP

ug/L = (PPMV) (molecular weight)

PPMV = uL per liter of gas

molecular weight = molecular weight of compound in grams

* 1 mole of gas occupies 22.4 liters at STP (0°C and 760mm Hg)

CONVERSION OF CONCENTRATION IN ug/1 @STP TO CONCENTRATION IN PPMV @STP

PPMV = (ug/L)(22.4*)molecular weight

PPMV = uL per liter of gas

molecular weight = molecular weight of compound in grams

* 1 mole of gas occupies 22.4 liters at STP (0°C and 760mm Hg)



CONVERSION CALCULATIONS FOR GAS ANALYSIS (CONT.)

CALCULATING % CHLORIDE OF A COMPOUND

% Chloride Content = <u>Total molecular weight of Cl in compound X100</u>

Total molecular weight of compound

CONVERSION OF ug/L REPORTED VALUE TO ug/l CHLORIDE AS CHLORIDE

ug/L Cl as Cl = (ug/L of compound)(% chloride content)
100

CALCULATING % FLUORIDE OF A COMPOUND

% fluoride Content = <u>Total molecular weight of F- in compound</u> X100

Total molecular weight of compound

CONVERSION OF uq/L REPORTED VALUE TO uq/l FLUORIDE AS FLUORIDE

ug/L F as F = (ug/L of compound)(% fluoride content)
100

CONVERSION OF ug/l F1- AS F- TO ug/l F- as C1-

ug/l F as Cl = (ug/l F- as F-) (molecular wt. of Cl-) molecular wt of F



CONSTANTS USED IN GAS CALCULATIONS

STP = 0°C AND 760mm Hg

0°C = 273° Kelvin

1 mole of gas occupies 22.4 liters at STP

1 mole = molecular weight of the compound in grams

1 cubic meter = 1000 liters

1 cubic foot = 28.31 liters

inches Hg X 25.4 = mm Hg

ppm = uL per liter = mL per cubic meter

ug/L = mg/cubic meter

Chloride molecular weight = 35.453 grams

Fluoride molecular weight = 18.998 grams

Bromide molecular weight = 79.904 grams

Iodide molecular weight = 126.904 grams

SAMPLE CALCULATION FOR REPORTED VALUE OF 200ppm FLUOROTRICHLOROMETHANE

ug/L = (200ppm)(137.37 molecular weight) = 1226 ug/L22.4

% Chloride Content = 106.36 grams chloride X 100 = 77.42% 137.37 grams molecular weight

 $ug/L Cl^{-} as Cl^{-} = (1226 ug/L) (77.42% Chloride) = 949 ug/L Cl^{-} as Cl^{-}$

% Fluoride Content = 18.998 grams fluoride X 100 = 13.83% 137.37 grams molecular weight

ug/l F- as F- = (1226 ug/l) (13.83% Fluoride) = 170 ug/l F- as F⁻ 100

 $ug/l F^- as Cl^- = (170 ug/l as F^-)(35.45) = 317 ug/l F^- as Cl^- 19.00$

ug/l Total Org. Halide as Cl ==

(949 ug/l as Cl-)+(317 ug/l F- as Cl-) = 1266 ug/l as Cl- (TOH/CL)



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RADIOCHEMISTRY LABORATORY

QUALITY ASSURANCE/QUALITY CONTROL PROGRAM

BARRINGER LABORATORIS INC.

15000 WEST 6TH AVENUE, SUITE 300

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(303) 277-1687

Prepared by: David B. Lasher

Rev. January, 1990

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1.0 INTRODUCTION

Increased public awareness of the radiation hazards associated with the uranium and nuclear industries has demanded a continuous program of monitoring the environment. The accurate and reproducible measurement of radioactivity in environmental materials is of utmost importance to any surveillance program related to these industries. There must be some degree of confidence associated with these measurements, and this confidence is determined and maintained by a good quality assurance/quality control program.

Barringer Laboratories' Radiochemistry Quality Assurance Program was developed to insure that our laboratory produces the highest quality data for its industrial and governmental sponsors. The program was designed to meet new regulatory requirements, and to take advantage of new technology.

Our Quality Assurance Program is based on the recommendations of the U.S. Nuclear Regulatory Commission, Office of Standards Development, Regulatory Guide 4.15 entitled "Quality Assurance for Radiochemical Monitoring Programs (Normal Operations) - Effluent Streams and the Environment (1977); and the EPA Manual for the Certification of Laboratories Analyzing Drinking Water (Criteria and Procedures Quality Assurance), EPA-570/9-82-002 (October 1982).

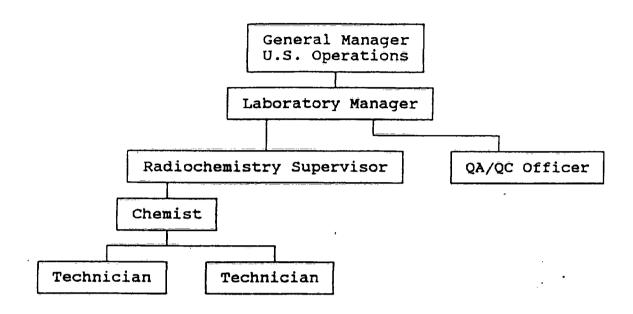
We have maintained our certification for all radiochemical parameters under the Safe Drinking Water Act since 1979.

The primary purpose of this quality assurance manual is to establish guidelines for the Radiochemical Laboratory to assure that analytical data being reported by this laboratory (1) are valid and are of known and acceptable precision and accuracy, (2) are representative, and (3) have the necessary documentation available in support of the analytical results.

The guidelines presented in this manual shall represent at least the minimum procedural requirements for our program and will address such areas as organization, personnel qualifications, sample handling, chain-of-custody, radiochemical procedures, Quality Control, data reduction, validation, and reporting protocols. They are designed to establish a reasonable and workable Quality Assurance program, one that is both efficient and reasonably comprehensive, so as to provide to our clients both quality service and analytical results which can be held with a high degree of confidence as to their accuracy and completeness of documentation.

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Organization



2.2 Responsibilities

Laboratory Manager - Responsible for the overall operation of the entire laboratory, including review of the QA/QC program for compliance by each division.

Radiochemistry Supervisor - Responsible for the operation of the Radiochemistry Laboratory. Responsibilities include; training of personnel, procedure development, data assessment, implementation of QA/QC procedures and maintenance protocols, workflow, identifying problem areas and implementing corrective action.

Chemist - To perform any and all analyses offered by the laboratory with proficiency. To assist the laboratory supervisor and provide technical assistance to the technicians. To report any procedural or analytical problems encountered in the laboratory to the supervisor.

Technician - To perform routine analyses with little or no supervision as specified by the supervisor. Must complete a training program and show proficiency with an assigned procedure before performing on a routine basis. Responsibility includes reporting any problems encountered in the laboratory to the immediate supervisor.

3.0 SAMPLE COLLECTION AND PRESERVATION

All samples sent to our laboratory are collected by the sponsor or his representative. Barringer Laboratories has the resources and expertise to consult with the client on sample site selection and collection methods, if needed. Geological and environmental consulting services are also available.

Sample preservation techniques must be used to insure that the sample is stabilized prior to being sent for analysis. The following procedures are recommended.

3.1 Air Particulates

To avoid loss of particulate material contained on filters, they should be folded once to conceal the exposed side of the paper and placed in an appropriately sized, labeled envelope.

3.2 Water/Wastewater

Waters and waste waters can be analyzed for total, dissolved and/or suspended radionuclides. If dissolved radionuclides are to be determined, the sample should be filtered on site using a 0.45 micron membrane filter and acidified with reagent grade concentrated nitric acid to pH 2. If both dissolved and suspended radionuclides are to be determined, the sample should be cooled and transported to the laboratory as quickly as possible without prefiltering and without any addition of acid.

If more than one radiochemical parameter is to be determined on a sample, we recommend shipment of at least one gallon to complete the usual suite of analyses (see Table I in the Appoendix for required sample volumes and respective LLD's). Plastic sample containers are preferred for shipment.

Samples which have not been filtered prior to the addition of HNO₃ cannot be determined on a dissolved and/or dissolved/ suspended basis. They can only be anlayzed on a "total" basis.

Samples should not be allowed to sit in direct sunlight where they might become hot. Conversely, samples should not be allowed to freeze.

3.3 Vegetation, Soils, and Sediments

These samples should be sealed in plastic bags and shipped "as is" immediately to the laboratory. The laboratory will do the necessary preparation prior to analysis. See Appendix for sample quantity and lower limit of detection requirements.

3.4 Animal Material

Animal material (fish, rodents, etc.) should be sealed in plastic bags and shipped frozen or on ice immediately to the laboratory for subsequent analysis.

3.5 Bioassay Samples

Urine samples should be acidified with concentrated reagent grade nitric acid to pH 2 prior to shipment. Plastic bottles or one (1) gallon cubitainers can be used for shipment.

4.0 SAMPLE HANDLING AND TRACKING

4.1 Sample Log-in

Upon receipt at the laboratory, samples are checked for damage, and for any discrepancies between the accompanying documentation, sample ID's, requested analysis, or any other pertinent information. If any discrepancies are found, the client is notified immediately. Each sample is assigned its own unique log number and a record of dates received, condition at receipt, client name/address, sample ID's, and requested analyses is made and this information is stored into the computer based laboratory information management system (LIMS), which allows for sample tracking, data storage and manipulation, and final reporting of the results of analysis.

4.2 Chain-of-Custody

After log-in is complete, samples are placed in a holding area until ready for analysis. Samples that require strict Chain-of Custody protocol are secured and tracked by a Chain-of-Custody record which follows each sample. Persons taking these samples from secured holding areas must sign and date the Chain-of-Custody record upon removal and return.

4.3 Data-Report Sheets

A data-report sheet is made for each set of requested analyses according to the assigned log number (any special handling instructions or procedures are noted on the data-report sheet). These sheets are given to the analyst responsible for performing the analysis. They tell him/her which samples need to be analyzed and for what. The final results are recorded onto these sheets by the analyst along with the dates of analysis, his/her name, duplicate results, and analysis quality control parameters.

5.0 LABORATORY SAMPLE PREPARATION

5.1 Water/Wastewater

Water and wastewater can be analyzed for various constituents on a dissolved, suspended, and/or total basis. Dissolved pertains to any constituent which passes through a 0.45 micron filter, and suspended to that which is retained by the filter. Total pertains to combined dissolved and suspended fractions, typically these samples are determined on an "as is" basis without any filtering.

Waters submitted for dissolved analysis should be filtered in the field and acidified to pH 2 with nitric acid, prior to shipment. No further laboratory preparation prior to analysis is required.

Those samples submitted for "total" analysis or for "dissolved and suspended" analysis should not be filtered or acidified.

These samples should be shipped on ice only, and should be kept under refrigeration while in the laboratory. Prior to analysis, the samples are mixed well to provide a homogeneous mixture and filtered through a 0.45 micron membrane filter. The suspended fraction is treated with a mixed acid digestion according to procedures as outlined in the procedure manual for the radionuclide of interest, and is carried through the procedure as specified.

Some waters with high suspended solids content are difficult to filter through a 0.45 micron filter while in the field and may require laboratory preparation and final filtration. UNLESS OTHERWISE SPECIFIED BY THE CLIENT, it is assumed that all analyses are on a dissolved basis and that the samples have been properly filtered and preserved prior to shipment. If it is apparent the samples have not been filtered prior to shipment, and the client has not specified whether the requested parameters are on a dissolved, suspended, or total basis, the samples will be analyzed "as is" and run on a total basis. This is to be noted on the report sheets for future reference.

5.2 Soils and Sediments

Soil and sediment samples are oven dried at 100 degrees centigrade to remove any moisture; passed through a jaw crusher and riffle splitter, and then pulverized to approximately 100 mesh.

5.3 Air Particulates, Vegetation, Animal and Fecal Material

Air particulates, filters, vegetation, and animal material are subjected to a mixed acid digestion of nitric, perchloric, and hydrofluoric acids to moist salts.

5.4 Urine Samples

Urine samples are preserved with nitric acid upon receipt at the laboratory, and are analyzed on a "total basis" according to procedures as outlined in the procedure manual for the radionuclide of interest.

6.0 ANALYTICAL PROCEDURES - OVERVIEW

All analysts are provided with copies of the procedures on which they are working. The procedures give an abstract, the concentration range that the procedure is applicable to, a list of possible interferences, directions for reagent preparation, safety considerations, a description of instrumentation set—up and use, and sample calculations.

6.1 Radium 226

Radium is coprecipitated from the sample with the addition of barium and lead sulfate. In addition, Barium-133 is added as a tracer to determine chemical yield. The final precipitate is mounted on a membrane filter and then counted in an alpha spectrometer for Ra-226 at 4.8 MeV. A NaI gamma spectrometer system is used to determine the amount of Ra-226/Ba-133 recovered.

Reference:

- 1. Standard Methods for the Examination of Water and
 Wastewater, 16th ed. (13th ed.), American Public Health
 Association, 1986, Method 705 (Method 304).
- 6.2 Isotopic Thorium (Th-228, Th-230, Th-232)

Thorium is collected on an ion exchange resin, eluted and mounted on a planchet. The planchet is then counted in an alpha spectrometer. Recovery is determined by addition of Thorium-227 or Thorium-229 tracer carried through the procedure.

Reference:

Handbook of Analytical Procedures, J.N. Latimer, et al, RMO-3008, USAEC Grand Junction Office, Analytical Laboratory, February, 1970.

6.3 Gross Alpha-Beta

The sample is mounted on a 2" diameter planchet where no more than 100 mg of solids from the sample is deposited and counted in an alpha/beta low-background proportional counter. Correction is made for self-absorption due to the solids present.

Reference:

- Prescribed Procedures for Measurements of Radioactivity in Drinking Water, H.L. Krieger, E.L. Whittaker, Environmental Protection Agency, EMSL-LV, EDA-600/4-80-777, August 1980, p. 1-49, Method 900.0.
- 2. Standard Methods for the Examination of Water and Wastewater, 16th ed., Method 703 (13th ed., Method 302)
 American Public health Association, 1986.

6.4 Lead 210

Lead is collected on an ion exchange resin, eluted, and precipitated as the chromate. The precipitate is mounted on a planchet and aged. The bismuth 210 daughter is then counted in a low-background proportional counter. Recovery is determined gravimetrically.

Reference:

Methods for Determination of Radioactive Substances in Water and Fluvial Sediments. Techniques of Water Resources Investigations of the U.S. Geological Survey, Chapter A5, 1977.

6.5 Polonium 210

Polonium is precipitated as the sulfide, then counted in a low-background gas-flow proportional counter.

Reference:

1. Quantitative Radiochemical Method for Determination of Major Sources of Natural Radioactivity in Ores and Minerals, N.J. Rosholt, Jr.

6.6 Radium 228

Radium 228 is determined by purification and subsequent beta counting of its daughter Actinium 228. Radium 228 is isolated by double precipitation of the sulfate using lead and barium sulfate as carriers. The barium sulfate is dissolved in diethylenetriaminepentaacetic acid (DTPA) and the Actinium 228 daughter is allowed to ingrow for at least 36 hours. The radium is then precipitated with BaSO₄ by the addition of acetic acid, and the supernatant containing the Actinium 228 is transferred to a separatory funnel for the extraction of the actinium into bis(2-ethylhexyl) phosphoric acid (HEDHP). The HDEHP is then back extracted with 1N nitric acid, and the aqueous phase is transferred directly onto a counting planchete, evaporated to dryness, and counted in a low-background beta counter for 60 minutes.

Reference:

- 1. A Procedure for the Determination of Radium 228, I.B. Brooks and R.L. Blanchard.
- Radium 228 Determination, D.R. Percival and D.B. Martin,
 Analytical Chemistry, Vol. 46, pg. 1742, (1974).

Prescribed Procedures for Measurement of Radioactivity in Drinking Water, H.L. Krieger, E.L. Whittaker, Environmental Protection Agency EMSL-LV, EPA-600/4-80-032, August, 1980, pq. 49.

6.7 Uranium

Natural uranium is determined by fluorimetry. Depending on the uranium concentration and possible interferences the sample can be run direct or can be carried through on extraction procedure prior to analysis. A NaF/LiF fusion is performed on the sample and the subsequent fusion pellet read in a fluorimeter.

References:

- 1. Microquantities of Uranium in Water by Fluorimetry, Annual Book of ASTM Standards, 1973, (D2907), 23.
- 2. Manual of Analytical Methods for the Uranium Concentrating Plant, Mines Branch Monograph 866, Dept. of Mines and Technical Surveys, Ottawa, Canada, (1959).
- 3. Environmental Measurements Procedures Manual, Harley, J.H., ed. 1972, HASL-300, U.S. Department of Energy, New York, N.Y.
- 6.8 Isotopic Uranium (U-234, U-235, U-238)

Uranium is coprecipitated with ferric hydroxide and separated from other radionuclides by anion exchange. Uranium 238, 234 and 235 are then determined by alpha-pulse height analysis.

Reference:

- Prescribed Procedures for Measurement of Radioactivity in Drinking Water, H.L. Krieger, E.L. Whittaker, Environmental Protection Agency EMSL-LV, EPA-600/4-80-032, August, 1980, pg. 49.
- Standard Method for the Examinatioan of Water and Wastewater, 16th ed., American Public Health Assoc., 1985.

7.0 INSTRUMENTATION

Alpha Spectrometer Systems:

- Six (6) Quad Canberra Model 7404 alpha spectrometers, coupled to Canberra Series 35 plus MCA.
- One (1) EG&G Ortec Model 576 alpha spectrometer, coupled to an Ino-Tech 5200 MCA.

Low-Background Proportional Counter Systems:

- One (1) Tennelec Model LB-4000 alpha-beta counting system with eight (8) detectors.
- One (1) Tennelec Model LB-5110 alpha-beta automatic counting system.
- Two (2) Canberra Model 7701 alpha-beta manual counters with associated electronics.

Gamma Spectroscopy Systems:

Four (4) NaI detectors coupled to a Canberra Series 35 MCA One (1) NaI detector coupled to an Ino-Tech Model 5200 MCA

Uranium Analysis:

- Two (2) Jarrell-Ash Fluorimeters
- One (1) EDA-Rotary Fusion Burner

8.0 LOWER LIMITS OF DETECTION (LLD)

Our Radiochemistry Laboratory can meet or exceed the LLD's as specified by the Nuclear Regulatory Commission (NRC) guide 4.14, in addition to any of the requirements specified by the EPA and the National Interim Primary Drinking Water Regulations (NIPDWR) (40 CFR 141 and 142).

Listed in the Appendix is a table of LLD's and minimum sample volumes required for each radionuclide to meet those LLD's. The LLD's are determined using the Nuclear Regulatory Commission (NRC) equation found in the NRC Regulatory Guide 4.14:

NRC LLD =
$$\frac{4.66 \text{ Sb}}{(3.7 \times 10^4) \text{ (E) (V) (Y)}} \times \frac{1}{e^{-7t}}$$

where:

Sb = standard deviation of the background (cps)

 $3.7 \times 10^4 = dps/mCi$

E = counting efficiency

V = volume (ml)

Y = yield

 τ = decay constant

t = time elapsed

e = 2.72

9.0 QUALITY CONTROL

Quality Control encompasses all of the procedures and laboratory practices that are in place to ensure the reliability and integrity of analytical results reported by the laboratory. It is a fundamental part of all quality assurance plans.

The following Quality Control guidelines have been established as part of our Quality Assurance program.

For monitoring precision and accuracy, each set of samples for each analysis is accompanied by:

- a) Blank
- b) Standard
- c) 10% Duplicates or Spikes

All standards used are NBS traceable. If a soil is being analyzed on a particular set, an additional soil standard is included.

Spikes are included whenever possible (sample volume permitting).

Participation in the EPA quarterly and monthly quality control cross check program is mandatory.

Duplicate and spiked field samples submitted by clients are encouraged.

In house "blind" standards and spikes are submitted on a regular basis.

Analytical balances are checked with class S weights prior to use. They are maintained under contract with a service company, cleaned and adjusted every six months.

Counting instruments are monitored for efficiency prior to each day's use and results are recorded in each logbook.

Instrument backgrounds are monitored on a regular basis.

Quality control charts are used and maintained.

Precision and accuracy data are documented.

9.1 Methods Monitoring

All methods and procedures for radiochemical analysis, presently in use by this laboratory, have been well documented as to their reliability and representativeness; most have been in use, extensively, for several years.

For purposes of monitoring methods and analyst error through precision and accuracy criteria, each set of samples for each analysis include a blank, standard, and 10% duplicates. Blind EPA or other unknown standards and duplicates may be included within a set.

All pertinent information including sample log-numbers, sample volumes, dates of analysis, blanks, standards, and duplicates are recorded onto the laboratory worksheets for each set of analyses. Final results, in addition to standard and duplicate precision and accuracy data, are calculated and recorded by the analyst onto these sheets, which are submitted to the laboratory supervisor for approval prior to reporting.

9.2 Accuracy Criteria

Standard and spike results must fall within a range of ±10% deviation from the known (taking into account a counting error of 2 sigma) to be acceptable.

9.3 Precision Criteria

Duplicate analyses must be within 10% deviation from the mean (taking into account a counting error of 2 sigma) to be acceptable.

9.4 Suspicious Data

Any standard or duplicate results that fall outside of the above ranges are suspect and must be rerun along with selected samples from the set to determine the extent of the problem. All further analyses are discontinued until the problem can be resolved.

Entire sets may have to be rerun if the problem appears to be non-isolated.

9.5 Instrument Monitoring

All instrumentation used for counting purposes are checked periodically for performance. Each type of instrument will have its own specific performance-check procedure. All are checked for background count rates and counting efficiencies on a regular basis. Each instrument has its own logbook where a history of background count rates, counting efficiencies, and maintenance performed has been recorded.

Control charts are used to monitor both the background count rate and counting efficiencies. Upper and lower warning and control limits have been established for each instrument through standard deviations derived from months of data. Two (2) standard deviations or 2 sigma delineate the upper and lower warning limits, while 3 sigma define the upper and lower control limits.

Low-Background Proportional Counters

These instruments are checked for counting efficiency prior to each day of use with an NBS traceable Th-230/Sr-90 source. The total alpha counts (or rate) and total beta counts (or rate) are recorded in the logbook, and are compared with the control charts to determine if they are within the previously set control limits. Anything above or below 3 sigma is considered out of control and the instrument is taken out of service until the problem is corrected.

Instrument background count rates are monitored on a weekly basis, and the results are recorded in the logbook. Control charts are also used to identify potential problems, with 3 sigma being defined as the upper and lower control limits.

Alpha Spectrometers

These instruments are sensitive to environmental changes and need to be monitored for counting efficiency and background count rates on a daily basis: Thorium 230 and Radium 226 standards are run before, during, and after each set of samples, to determine if the detectors and instrument are working properly. In addition, each alpha spectrometer has an internal calibrated test-pulse, vacuum and detector leakage-current display, and dead-time display to facilitate identification of instrument problems.

Gamma Scintillation Counters

Prior to their use a Barium 133, or Cesium 137 (or both) standard is counted to determine counting efficiency. Control charts are used to help identify trends or potential problems that could lead to erroneous data. This information along with background count rates are recorded and maintained in the instrument logbook.

10.0 DATA REDUCTION, VALIDATION AND REPORTING

Each analyst is responsible for doing his or her own calculations and reporting the final results on their laboratory worksheets. These worksheets contain all pertinent information and include the blank, standard, and duplicate results. Calculations are performed using pre-programmed computer cards.

The worksheets and final results are submitted to the laboratory supervisor for approval. The results are checked for discrepancies in standards, blanks, and duplicates or errors in calculation.

Once approved the worksheets are signed and the results are submitted for typing of the final report. The final report is checked by the laboratory supervisor for accuracy and signed.

Accompanying each final report is a copy of the Quality Control Sheet which lists dates of analysis, analysts name, standard and duplicate results, and references.

11.0 PERFORMANCE AND SYSTEM AUDITS

It is the responsibility of the QA Officer to perform audits of the laboratory and quality control procedures on a regular basis, to assure these procedures are being followed and the necessary documentation maintained and kept up to date. These audits shall be performed on a quarterly basis, and the results shall be maintained in the QA Officers audit notebook.

The audit shall include (1) a check of the laboratory worksheets for blank, standard, and duplicate results, (2) a check of the counting equipment and instrument log-books for proper calibration procedures and maintenance protocols, and (3) a check of the monthly EPA cross-check results.

12.0 CORRECTION ACTION

The laboratory supervisor is to be notified immediately of any problems or discrepancies encountered during any part of the analytical process. It is his or her responsibility to initiate corrective action to resolve those problems as soon as possible.

The procedures as outlined in this manual are designed to minimize the chances for error, and to identify problems or potential problems before they have had the opportunity to do significant harm to the quality of data. Some corrective measures have been addressed in the previous sections of this manual. More serious problems dealing with instrument or system calibrations may have to be addressed separately, referring to instrument manuals or other outside sources.

13.0 QA REPORTS TO MANAGEMENT

The results of the quarterly performance and system audits by the QA Officer are to be submitted to both the laboratory supervisor and upper level management for their evaluation.

APPENDIX I

JOB DESCRIPTIONS

JOB DESCRIPTION FOR LABORATORY SUPERVISOR

FUNCTION: To supervise the overall operation of the laboratory.

RESPONSIBILITIES:

- 1. To assign priorities to analytical jobs and other tasks at the lab.
- To make daily work assignments to laboratory personnel and perform analyses as necessary.
- 3. To provide technical assistance and training for all other laboratory personnel.
- 4. To insure that all equipment is correctly calibrated and maintained.
- 5. To insure that all reports are carefully inspected and all data meet or exceed standards for acceptable quality.
- 6. To maintain a working knowledge of all analytical methods in use at the lab.
- 7. To be knowledgeable in all analytical procedures in use at the lab and assist in solving problems associated with their use.
- 8. To provide technical assistance to clients and maintain their goodwill.
- To examine, select, or in other ways assist in the development of new laboratory procedures.
- 10. To monitor work flow and insure that analytical jobs and other tasks are completed on time.

- 11. To provide technical assistance to customers.
- 12. Other responsibilities as assigned.

JOB DESCRIPTION FOR CHEMIST

FUNCTION: To perform routine and nonroutine analyses with little or no supervision.

RESPONSIBILITIES:

- 1. To rapidly and accurately complete daily work assignments.
- 2. To maintain a clean, safe and orderly work area.
- To provide technical help and training for Technicians of all levels.
- 4. To help calibrate and maintain laboratory equipment in good working order.
- 5. To monitor chemical and supply levels to insure adequate quantities to complete work assignments.
- 6. To review selected lab data for accuracy and quality and to perform check analyses where necessary.
- 7. To further seek qualified help if procedures and/or results are subject to question.
- 8. To follow routine analytical procedures and solve problems associated with their use.
- 9. To examine, select, or in other ways assist in the development of new laboratory procedures as assigned.
- 10. Other responsibilities as assigned.

JOB DESCRIPTION FOR LAB TECHNICIAN

FUNCTION: To perform analytical assignments under general supervision.

RESPONSIBILITIES:

- 1. To rapidly and accurately complete daily work assignments.
- 2. To maintain a clean, orderly and safe work area.
- 3. To seek qualified help if procedures and/or analytical results are subject to question.
- 4. To maintain assigned laboratory equipment in good working order and to notify the appropriate staff member that equipment repair is necessary.
- 5. To maintain adequate quantities of chemicals and supplies necessary to complete daily assignments and to notify designated staff member when supply orders are required.
- 6. Other assignments as required.

APPENDIX II

LOWER LIMITS OF DETECTION

Radiochemistry Lower Limits of Detection (LLD)*

Sample		(Ref)	Minimum Sample	
Type	<u>Analysis</u>	Methods**	Size	<u>LI,D</u>
Water	Radium-226 Thorium-230 Lead-210 Polonium-210 Uranium-Nat Radium-228 Gross Alpha Gross Beta Uranium-238/234 Cesium Gamma Emitters Radioactive Iodine	705/304(4) (11) (5) (6) D2907(9) (12)(13) 900.0(14) 900.0(14) 908.0(14) 901.0(14) 901.1(14) 902.0(14)	1.0 liter 0.5 liter 0.5 liter 0.5 liter 0.1 liter 1.0 liter 0.2 liter 1.0 liter 1.0 liter 1.0 liter	0.2 pCi/l 0.2 pCi/l 1.0 pCi/l 1.0 pCi/l 0.2 pCi/l 0.9 pCi/l 1 pCi/l 2 pCi/l 0.2 pCi/l 10 pCi/l 10 pCi/l 10 pCi/l
	SR90/SR89 Tritium	905.0(14) 906.0(14)	1.0 liter 1.0 liter	1 pCi/l 1000 pCi/l
Air Particu- lates	Radium-226 Thorium-230 Lead-210 Polonium-210 Uranium-Nat Uranium-238/234 Gross Alpha Gross Beta	704/304(4) (11) (5) (6) D2907(9) 908.0(14) 900.0(14)	2x10 ⁸ liters 2x10 ⁵ liters 5x10 ⁵ liters 5x10 ⁸ liters 2x10 ⁸ liters 2x10 ⁸ liters	1x10 ⁻⁷ pci/1 1x10 ⁻⁶ pci/1 2x10 ⁻⁶ pci/1 2x10 ⁻⁷ pci/1 1x10 ⁻⁷ pci/1 1x10 ⁻⁷ pci/1
Soil	Radium-226 Thorium-230 Lead-210 Polonium-210 Uranium-Nat Uranium-238/234 Gross Alpha Gross Beta	705/304(4) (11) (5) (6) D2907(9) 908.0(14) 900.0(14) 908.0(14)	1.0 grams 1.0 grams 2.0 grams 1.0 grams 1.0 grams 1.0 grams	0.2 pCi/g 0.2 pCi/g 0.2 pCi/g 1.0 pCi/g 0.2 pCi/g 0.2 pCi/g
Vegetation	Radium-226 Thorium-230 Lead-210 Polonium-210 Uranium-Nat Uranium-238/234	705/304(4) (11) (5) (6) D2907(9) 908.0(14)	4 kilograms 1 kilogram 1 kilogram 1 kilogram 1 Kilogram 1 kilogram	5x10 ⁻⁵ pCi/g 2x10 ⁻³ pCi/g 1x10 ⁻³ pCi/g 1x10 ⁻⁴ pCi/g 2x10 ⁻⁴ pCi/g 2x10
Urine	Radium-226 Thorium-230 Uranium-Nat	705/304(4) (11) D2907(9)	1 liter 2 liters 0.1 liter	0.2 pCi/l 0.1 pCi/l 1.0 pCi/l

^{*} As determined by the LLD calculation found in the NRC Regulatory Guide 4.14

^{**} For a complete list of references and corresponding method numbers see the attached Methods Reference Sheet.

ENVIRONMENTAL LABORATORY METHODS REFERENCE SHEET

- 1. Flameless A.A., <u>Methods for Chemical Analysis of Water and Wastes, 1980.</u> Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268 March 1983.
- Flame A.A., <u>Methods for Chemical Analysis of Water and Wastes</u>, 1980. Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, March 1983.
- 3. <u>Methods for Chemical Analysis of Water and Wastes</u>, 1980. Environmental Monitoring and Support Laboratory. Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, March 1983.
- 4. Standard Methods for the Examination of Water and Wastewater, 14th ed., American Public Health Assoc., 1975.
- 5. <u>Methods for Determination of Radioactive Substances in Water and Fluvial Sediments.</u> Techniques of Water Resources
 Investigations of the U.S. Geological Survey, Chapter A5, 1977.
- 6. Quantitative Radiochemical Method for Determination of Major
 Sources of Natural Radioactivity in Ores and Minerals. p. 1307,
 ACS. Aug. 1954,; and p. 1398, AGS, Oct. 1957, N.J. Rosholt, Jr.
- 7. The Determination of Radium-226 in Uranium Ores and Mill Products by Alpha Energy Spectrometry. Canmet Report 76-11, Canadian Centre for Mineral and Energy Technology.

- 8. Analytical Method for the Determination of Polonium 210.

 Thorium 230 in Uranium Ore. Concentrates, and Plant Liquors, PG report 356(s), United Kingdom Atomic Energy Authority.
- 9. Microquantities of Uranium in Water by Fluorimetry. Annual Book of ASTM Standards, 1973, (D2907) 23.
- 10. Manual of Analytical Methods for the Uranium Concentrating

 Plant, Mines Branch Monograph 866, Dept. of Mines and Technical
 Surveys, Ottawa, Canada, (1959).
- 11. <u>Handbook of Analytical Procedures</u>, J.M. Latimer, et al, ROM-3008, USAEC, Grand Junction Office, Analytical Laboratory, February, 1970.
- 12. Procedure for the Determination of Radium-228, I.B. Brooks and R.L. Blanchard.
- 13. Radium-228 Determination, D.R. Percival and D.B. Martin, Analytical Chemistry, vol. 46, pg. 1742 (1974).
- 14. Prescribed Procedures for Measurements of Radioactivity in Drinking Water, H.L. Krieger, E.L. Whittaker, Environmental Protection Agency, EMSL-LV, EPA-600/4-80-777 August 1980, p.1-49.
- 15. Environmental Measurements Procedures Manual, Harley, J.H., ed. 1972. HASL-300, U.S. Department of Energy, New York, N.Y.

APPENDIX B

Standard Forms and Typical Diagrams



SITE SUPERVISORS DAILY REPORT

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°F, °C @ hr.	Sleet	
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Field Project Debriefing Form

Project Name: PH I RI - PASCO LANDFILL	Project Manager:	DAVE HADDOCK	
Project No.: 624419/7020	Field Supervisor:	CRAIG MAXEINER	
Phase/Task No.:	Completed By:		
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BURLINGTON ENVIRONMENTAL INC.

Field Project Debriefing Form

Project No.: 624419 Task No.(s):
5. Scope of Work: Rating
6. Health and Safety: Rating
7. Documentation: Rating
8. Mathes Personnel: Rating
9. Subcontractor Personnel: Rating

BURLINGTON ENVIRONMENTAL INC.

Field Project Debriefing Form

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BURLINGTON ENVIRONMENTAL INC. Field Project Debriefing Form

Proj	ect No.:	624419		Task No.(s):
15.	Suggestio	ns and	Additional	Comments:
				
	 			
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SITE EXIT CHECKLIST

Project No.: 624419 Phase 7020 Task

Date: F	orm Seria	al No.:	SEC	
Project Name: PH I RI - PASCO LANDFILL				
		YES	<u>NO</u>	<u>N/A</u>
Have bumper posts been set?	• • • • • • •			
Have concrete pads been installed around the wel	ls?			
Have bumper posts and wells been painted?				
Are wells locked?	• • • • • •			
Have wells been labeled?	• • • • • • •		=	
Have wells been photographed?	• • • • • •			
Have wells been measured?				
Have a final round of water levels been taken?	• • • • • •			
Has a final round of well air quality been taken	1?			
Have drill sites been cleaned up?				
Have drums been moved?				
Have drums been staged?				
Are drums labeled?				
Are drums sealed?				
Has drum disposal been arranged?				
Has all equipment been decontaminated?				
Has all "hot" equipment been disposed of?				
Has all equipment been loaded?				
Has all rental equipment been returned?				
Is all equipment ready for travel?				
Have all subcontractors been notified at end of	job?	***		
Have utilities been shut off?				
Have wells been surveyed?				
Have well caps been notched (not on flush mount)?			
Have weep holes been drilled in well protectors	?			
Other:				
Signature:				

Project	Name	PH_I_RI	PASCO LANDFILL	
Project	No	674419	Phase 6020	Task



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Project	No.	624419	Phase 6020	Task	



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Project	Name	PH I RI -	PASCO LANDFILL	
Project	No	674419	Phage 6020	Task



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Project Name	PH I RI -	PASCO LANDFILL	
Project No.	624419	Phase 6020	Task



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Project	Name_	PH I RI	- PASCO LANDFILL	
Pronect	No.	624419	Phase 6020	Task



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SAMPLE CONTROL LOG

Serial	No.	SCL	
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BURLINGTON SAMPLE NUMBER	LABORATORY SAMPLE NUMBER	C.O.C. LOG NUMBER	BILL OF LADING NUMBER	DATE SHIPPED	CUSTODIAN'S NAME	LABORATORY NAME	DATE ANALYSIS PERFORMED	DATE RESULTS RECEIVED
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210 West Sand Bank Fload P O. Box 330 Culumbia, II. 62236-0330 618/281-7173 618/281-5120 FAX

CHAIN-OF-CUSTODY RECORD

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BURLINGTON ENVIRONMENTAL INC.

DRUM LOG

Date:	Time: Job No:_6	24419 Phase: 6020 Task:
Drum No:	Sample No:	Sample Control No:
DRUM SIZE:	DRUM TYPE:	
Unknown	Unknown	
55 gal	Metal	
30 gal	Plastic	
Other	Fiber	
Specify	Specify	• • • • • • • • • • • • • • • • • • • •
DRUM COLOR:	Drum Marking Key	Word 1
Unknown		
Primary	Drum Marking Key	Word 2
Secondary		
	Drum Marking Key	
•		
DRUM CONTENTS:	Drum Contents Co	olor:
Unknown	DRUM AMOUNT:	
Liquid		
Sludge	Unknown	
Gas	Full	
Trash	Part, Inches_	
Jirt -	Empty	
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THE CONTENTS OF THIS DRUM WERE SAMPLED BY BURLINGTON ENVIRONMENTAL INC. SEATTLE, WASHINGTON (206)767-3306
SAMPLE ID:
SAMPLER: (SIGN HERE)
DATE:

BURLINGTON	PROJECT NAME PH I RI - PASCO LANDFILL
ENVIRONMENTAL	AND DEL MARIE CO C. NO. C. CONTROL CON
•	FROJECT NO. 624419 FHASE 7020 TASK
	SUPERVISOR DATE
DAILY LOG	WEATHER
DAILT LOG	CONTRACTORS ON SITE
	REVIEWED BY
e following was noted:	
	
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FIELD REPORT



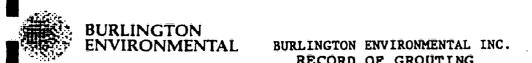
RECORD OF SUBSURFACE EXPLORATION

Page of Borehole No. Well No.

		NAME	. F	H I RI - PASCO LA	DFILL								PROJECT NO: 624419	İ
PROJECT NAME: PH I RI - PASCO LANDFILL ELEVATION: BORE-CLE LOCATION/CCORDINATES: .														
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224410						We	il Compi	etion Report
Project No. 624419 Phase 6020 Task			Cou	uty	·		Weil #	·
				Grid Cod	rdinate: North	ing		Easting
Drilling Contractor:	<u>.</u>	. 			Da	te Drilled	Start:	
								opleted:
F 1 1-1								•
Drilling Method:						rimar ichb	9/:	
Annular Space Detail	5					Ele	vations	— .01 ft. _ MSL Top of Protective Casin
Type of Surface Seal:		· 		-		I =		MSL Top of Riser Pipe ft. Casing Stickup
Type of Annuar Sesiant:				-	TYT	T		
Amount of comens: # of	bags	lbs. p	er ber	-		<u> </u>		MSL Ground Surface 1t. Top of annular sealant
Amount of bentomus: #	of bags	lbs. p	er sag	-				
Type of Bentonite Seal (Gran	vier. Pailet): 		-	77.70	7721		
Amount of bencomics: = of Ba	ga	· · · · · · · · · · · · · · · · · · ·	lba. per bäg	-	Transcription of the second	Niedkirzieskowy		-
Type of Sand Pack:								
Source of Sand:								
Amount of Sand: # of ba								
Well Construction Ma	torials							
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	1 >	Type	r Type	E				
	Stainless Steel Specify Typ	Teflen Specify	PVC Specify	Other Specify Type				
Riser coupling joint	01 01 21	:- 07	1	337				
Riser pipe above w.L	i							
Riser pipe below w.t.						}		
Screea			<u> </u>	ļ				
Coupling joint screen to rise	r				1 1]]		
Protective casing	<u> </u>			<u> </u>				
Measurements	U	.01 ft (wi	here applica	ıbie)		₩ -		_ (L Top of Seal
Riser pape length	i			· — · · · · · · · · · · · · · · · · · ·		₩ -		_ (t. Total Seal Interval
Protective casing length			<u>.</u>			器 —		_ (t. Top of Sand
Screen length								
Bottom of screen to end cap	1				:/-			It. Top of Screen
Too of screen to first joint								
Total length of casing	İ					- (:		A Total Screen Interval
Screen slot size	<u> </u>	<u> </u>			<u> </u>			
a of openings in screen] ::		
Diameter of borehole (in)	<u> </u>							_ (L Bottom of Screen
(ID) of riser pipe (in)	<u> </u>					<u></u>		(L Bottom of Borehole
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SERIAL	NO.	GRT

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RECORD OF GROUTING

PROJECT NAME PH I RI - PASCO LANDFILL PROJECT NUMBER 624419	
PHASE 6020 TASK DATE	
TECHNICIANS WELL NO.	·
TIME OF GROUTING (HRS) FROMTO	
GROUT PUMP	
GROUT MIX PROPORTIONS CEMENT WATER BENTONITE	
PRE-GROUTING INFORMATION	
DEPTH TO WATER (Feet) REFERENCE POINT	
DEPTH TO BOTTOM OF WELL (Feet) REFERENCE POINT	
WELL DIAMETER (Inches) WELL VOLUME ft ³ GAI	<u>.</u>
COMMENTS	
GROUTING INFORMATION	
INITIAL VOLUME GROUT PUMPED (Gallons)	
GROUT TAKE AFTER PRESSURIZATION	
INCREMENT TÎME INITIAL FINAL VOLUM NUMBER INTERVAL (min) PRESSURE (PSI) PRESSURE (PSI) GROUT ADI	
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COMMENTS	

SERIAL NO. DAL - H20

PROJECT MAME PH I RI - PASCO LANDFILL

FROJECT NO. 624419 FRASE 7020 TASK

	BURLINGTON ENVIRONMENTAL
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	SUPERVISOR	DATE
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WELL DEVELOPMENT & FURGING GENERAL DATA

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PROJECT NAME PH I RI -	PASCO LANDFILL	· · · · · · · · · · · · · · · · · · ·	we	и. ис	
PROJECT NO. 624419					
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	WE:	. CONSTRUCTION			
דסדאב פובדה (הדו		SCREHCLE CA	AWETER (180	<u></u>
GRAVEL PACK INTERVAL (F	7)	WELL DIAMET	ia ikstot	E (120)	
WELL PROTESTOR:Y	45 KC	PACLOCK NO.			····
מטאאדדדר פה הנטום ואיבסו	אנננואס סאנאטס כפי	פ (פאנבאצ)			
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DATE OF MEASUREMENT _		TEN		WATER YO	
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WATER LEVEL INSTRUMENT		WEL CASIN			
INITIAL WATER LEVEL (FT)_	. <u></u>	GRAVEL PAC			
Linear feet of water			IES ;		·
LINEAR FEET SATURATED G	RAYEL PACK	TOTAL			
WATER QUALITY MEASUREM WELL VOLUME (ANNULUS) (WATER VOLUME TO SE REM WOTE: DEVELOPMENT IS TO DEVELOPMENT PLAN	GAL) MIN OVED (GAL) MIN BE PERFORMED IN	WELL CASING YOU	MAX	MUMD	
	WATER QUA	ULITY INSTRUMENTS			
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COMMENTS ____

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- 2. ANY INSTRUMENTATION CALIBRATION OR USE ANOMALIES SHOULD BE MOTED.
- T. APPEARANCE SHOULD BE NOTED BEFORE DURING AND AFTER DEVELOPMENT.



WATER SAMPLING DATA

SERIAL NO. WS_

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PROJECT N	PH I RI - PA	SCO LANDFILL		CR EGR	ING/WELL NO.
PROJECT N	c. 624419		Phase	7020	7.25K
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	•	WATER GU.	ALITY INSTRUM	EHT3	
5473	INSTRUMENT	SZRIAL XC.	CALIBRATION	7204	COMMENTS
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BURLINGTON ENVIRONMENTAL		FROJECT NAME PH I RI - PASCO LANDFILL				
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GENERAL SAMPLING

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ROJECT N	0	624419	AND	 .		70	20 TA	SK
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SAMPLE N	0.	SAMPLE LABEL NO		TIME	COLLEC	E	SAMPLE	DESCRIPTION
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TYPE OI	NT	SERIAL		ACTUAL RI CONCENTE	ADING/		LOCATION OF	F READING
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BURLINGTON ENVIRONMENTAL

SOIL/SEDIMENT SAMPLING

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ROJECT	Жид	PH I RI PASCO LA	NDFILL	Sample	LOCATION	NO
COJECT	NO	624419		PHASE (_	7020	TASK
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WATER LEVEL DATA

Project Title _ Monitor Well _	Project No. 624419 Phase No. 7020 Task No.							
	Land Surface Elev M.P. Elev							
Date	Time	WIL S.M.P.	WIL.	W1_ Elev.				
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BURLINGTON ENVIRONMENTAL INC.

TE EXCAVATED	R	PROJECT NUMBER 62441	.9 PHASE 7020 METHOD
DEPTH -FEET-	SAMPLE NUMBER	DESCRIPTION	COMMENTS
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NOWGS\FORMS\TPITLOG (REV 7/90)

GEOLOGIST SIGNATURE



LAGOON LIQUID / SLUDGE LOG

BURLINGTON ENVIRONMENTAL INC.

PROJECT NO. 624419	PHASE NO. 7020	TASK	LOCATION
CONTRACT NO	BORING NO.		
DATE DRILLED		PROJECT PH	I RI - PASCO LANDFILL
METHOD		OPERATOR	

DEPTH -FEET- SAMPLE INTERVAL/ SAMPLE NUMBER MATERIALS DESCRIPTION REMARKS REMARKS REMARKS			==	
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ENVIRONMENTAL				
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DAILY LOG	CONTRACTORS ON SITE			
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RECON SYSTEM PROBE SAMPLING WORK SHEET

PROJECT NAME:	PH I RI - PA	ASCO LANDFILL		DATE:	
PROJECT NO.:	624419			PHASE:	6020 TASK:
FIELD CREW:		NAME	<u> </u>		POSITION/DUTIES

CLIENT/SITE RE	PRESENTATI				
Probe Hole Number	Sample Number	Sample Depth	DUP	VAC	Comments
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BURLINGTON ENVIRONMENTAL INC. RECON® System

Analytical Parameter Set-up Sheet

DATE	JOB	NAME	PH I RI -	PASCO L	ANDFILL	
JOB NO. 624419	ANAI	LYTICAL	TECH _			
PHASE 6020 TASK		÷				
GC <u>Hewlett Packard 5890</u>						
DETECTOR FID TEMP PID TEMP ECD TEMP	(°C) (°C) (°C)					
INJECTOR TEMP (°C)						
COLUMN(S) DB-624/30 METER DB-5 /30 METER SE-54 /15 METER	0.53		Carrier Column E	Gas H Pressu	l, He . ire (kPa) _	
OVEN TEMPERATURE PROFILE	(°C)					
INITIAL TEMP 1	_ INI	TIAL T	IME 1 _		(mins.)	
RATE °C/min						
FINAL TEMP	FINAL	TIME _				
TOTAL ANALYSIS TIME	(mi	ns.)				
CHĒMĪCĀLS ANĀLYZĒD						
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RECON® SAMPLE ANALYSIS

Page

SERIAL NO. RCNC

of

Work Sheet

DATE _		PROJECT NO.	624419	JOB NAME PH I RI - PASCO LANDFILL
DHACE	6020	TASK		

Sample I.D.	Probe Hole Number	Depth (ft.)	Analysis Time	Inj. Vol. (µ1)	Multiplier	VAC in. Hg	Comments
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D = duplicate analysis QC = quality control



Date _____

RECON® SAMPLE ANALYSIS

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SERIAL NO. RCND

Data Summary Table

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Data QA:	
Date:	

OC quality control

µg/L micrograms of compound detected per liter of headspace vapor analyzed

	BURLINGTON ENVIRONMENTAL
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	SERIAL NO. DAL - PUMP					
BURLINGTON ENVIRONMENTAL	FROJECT NAME PH I RI - PASCO LANDFILL	FROJECT NAME PH I RI - PASCO LANDFILL				
Shirk - ELAA LIZOLAIALELA LWF						
	FRASE 7020 TASE	Κ				
	SUPERVISOR DATE					
DAILY LOG	WEATHER					
DAILLEOG	CONTRACTORS ON SITE					
	REVIEWED BY					
	FAGE					
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FIELD REPORT



HYDROGEOLOGIC PUMP TEST DATA

PROJECT NAME: PH I RI - PASCO LANDFILL

PROJECT NO: 624419 PHASE:7020 TASK

BASELINE

DATE	TIME	PUMP WELL #	LOG#	MW.#	MW. #	MW.#	MW.#	COMMENTS
							1	START TIME :
				OBS	ERVED READ	INGS		STOP TIME :

DATE	TIME	PUMP WELL #	LOG#	MW.#	MW. #	MW.#	MW.#	COMMENTS
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TOTALIZER COUNT GALLONS REMOVED

PG.	OF	
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PROJECT NAME: PH I RI - PASCO LANDFILL PROJECT NO. 624419 PHASE: 7020	TASK	DATE:
INITIAL TOTALIZER CT :		START TIME:

TIME	TOTALIZER CT.	FLOW G.P.M	COMMENTS
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	TIME	TIME TOTALIZER CT.	TIME TOTALIZER CT. FLOW G.P.M

PROJECT NUMBE	A:	PROJEC	T NAME:			AH MONITORING DATA SHEET PHASE# TASK#						
OCATION:			AMPLES COLLE	CTED:			SPONDING C	.O.C. #:				
		PURPOS			BACKGROUND		REMEDIATI		PERSONA	<u>L()</u>		
SAMPLE NUMBER	INSTRUMENT TYPE & NUMBER	CALIBI OR PRE	RATION DATA & POST SPAN	CALIBRATION. INSTRUMENT/ OR GAS USED	SAMPLE DURATIO		SAMPLE VOLUME	11 '	ATION OR	TYPE OF ANALYSIS REQUESTED		ULT/ IITS
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	3. CIRCLE APPI 4. PRINT OR TY			JE		SIGNAI	UNE	FRINTE	D NAME	DATESTIME	PAIDI	ar(nrt)
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RECON® SAMPLE ANALYSIS

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ERIAL NO. RCND	01

Data Summary Table

Date				
Project No	624419	Phase _	6020	Task

Semple I.D.	Probe Hole Number	Depth (Feet)	1,1-DCE	1,1-DCA	trans-1,2- DOE well	chloroform	1,1,1-TCA	Comments
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Data QA:	
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Date	:		

OC quality control

µg/L micrograms of compound detected per liter of headspace vapor analyzed



624419

Dhaca 6020 Task

Date

Droiget No.

RECON® SAMPLE ANALYSIS

Page ___ of

SERIAL NO. RCND

02

Data Summary Table

Sample I.D.	Probe Hole Number	Depth (Feet)	TCE	PCE www.	toluene	(Jug/L)	(ኮይ/ር)	Commente
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Data C	A:	
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Date:	
vale.	

OC quality control

µg/li micrograms of compound detected per liter of headspace vapor analyzed:

APPENDIX C

Generator's Waste Material Profile Sheet

GENERATOR'S WASTE MATERIAL PROFILE SHEET WORKSET

Return t	he con	pleted	profile	and	chai	n of custo	dy recard	to:
BURL!!	NGTON	ENVIR	CNME	NTAL	- C	HEMPRO D	DIVISION	
	2203 A	URPOR	T WAY	' SO.	•	SUITE 400	0	
	Si	ATTLS	. WAS	HING	TON	1 98134		
	(2	:C6) C-H	H-E-M-	P-R-C	(24	3-6776)		

GENERAL INSTRUCTIONS

This workset contains two forms:

- GENERATOR'S WASTE MATERIAL PROFILE SHEET
- GENERATOR'S CHAIN OF CUSTODY RECORD
- 1. The Generator's Waste Material Profile Sheet is a two-sided form. Both sides must be completed.
- 2. This document is perforated so the forms and instructions may be separated for your convenience. If the forms are separated, take special precautions to ensure that they are used to describe and identify ONLY the same waste.
- 3. Shaded areas on the forms are for CHEMPRO DIVISION use only.
- 4. All questions must be answered. A response of "none" or "NA" may be used if appropriate.
- 5. Answers must be printed in ink or typed.
- 6. Instructions are included to help you complete these forms correctly. The letters and numbers which precede each instruction refer to the lettered and numbered entries on the forms.
- 7. Both the Generator's Waste Material Profile Sheet and the Generator's Certification of Representative Sample forms must be signed.
- 8. The peel-off label must be completed before removal from the Chain of Custody Record and applied to the container which actually holds the sample material not on the shipping carton even if the sample already has another label.
- 9. If you have any questions concerning the use of these forms, please contact your CHEMPRO DIVISION Sales Representative.
- 10. MAKE A COPY OF THESE FORMS FOR YOUR RECORDS. SEND THE ORIGINALS AND ALL ATTACHMENTS TO THE ADDRESS SHOWN ABOVE OR TO THE ADDRESS PROVIDED BY YOUR CHEMPRO DIVISION SALES REPRESENTATIVE.

GENERATOR'S WASTE MATERIAL PROFILE SHEET

This information is required for a waste to be considered for transportation, treatment, storage or disposal. It is used to determine if the waste may be transported, treated, stored or disposed in a legal, safe, and environmentally sound manner.

ANSWERS MUST BE MADE TO ALL QUESTIONS and must be printed in inx or typed. A response of "NONE", or "NA" may be used if appropriate.

PART A. GENERATOR INFORMATION

- 1. GENERATOR NAME Enter the name of the generating facility.
- 2. GENEPATOR USEPA ID Enter the twelve character alpha-numeric descriptor issued by the USEPA to the facility generating the waste.
- 3. FACILITY ADDRESS Enter the street aggress (not P C. Box) of the generating facility.
- 4. GENERATOR CONTACT. Enter the name of a person who is handling disposal and who can answer questions accut the waste.
- 5. TITLE Enter generator contact's title.
- 6. PHONE Enter generator contact s telephone number.
- CONSULTANT Indicate the namets) of the individualist representing any consultant company you may be employing for the
 management of this particular wastestream with whom Chemorô Division will be communicating.
- 8. COMPANY Indicate which company is represented by any consultant(s) listed in Part A. 7.
- 9. PHONE Provide the phone number for any consultant which was listed in Part A. 7.

PART 3. MAIL BURLINGTON ENVIRONMENTAL - CHEMPRO DIVISION INVOICES TO:

- 1. If you want the invoice mailed to the same address as in PART A, check "Generating Facility". If you want the invoices mailed eisewhere, then indicate the name, phone, and address, as shown in numbers 2 through 5.
- 2. COMPANY NAME Enter the name of the company to which you want the invoices sent.
- 3. PHONE Enter the telephone number of the company to which you want the invoices sent.
- 4. ADDRESS Enter the address of the company to which you want the invoices sent.
- 5. ATTENTION: Enter name of person to whom invoices should be directed.

PART C. WASTE INFORMATION

- NAME OF WASTE Enter a name that is generally descriptive of this waste (e.g., cyanide plating waste, paint sludge, FC3
 contaminated dirt, still bottoms, wastewater treatment sludge).
- 2. PROCESS GENERATING WASTE List the specific process/operation or source that generates the waste (e.g., metal platting operation, paint spray booth, PC8 spill, solvent recovery, wastewater treatment plant).
- 3. ADDITIONAL INFORMATION Indicate if you have included a sample of your waste, a Material Safety Data Sheet (MSDS), or any analysis of your waste with this profile by checking the applicable boxless.

PART D. PHYSICAL CHARACTERISTICS OF WASTE

- 1. CCLCR Describe the color of the waste (e.g., blue, clear, varies).
- 2. PHYSICAL STATE . If the four boxes provided do not apoly, a descriptive phrase may be entered after "Other" (e.g., gas).
- LAYERS Check all applicable boxes. Multi-layered means more than two layers (e.g., oil/water/sludge). Bi-layered means the waste is combrised of two layers which may or may not be of the same phase (e.g., oil/water, solvent/sludge). Single phased means the waste is homogeneous.
- 4. SPECIFIC GRAVITY Indicate the range. The specific gravity of water is 1.0. Most organics have a specific gravity less than 1.0 (i.e., they float in water). Chlorinated solvents, most inorganics and paint studge have specific gravities greater than 1.0 (i.e., they sink in water).
- 5. FREE LIQUIDS Check "YES" if liquid is usually present when packaging for shipment and estimate the percent of liquid volume. Check "NO" if there are no free liquids as defined by the Paint Filter Test (SW 846 Method 9095).
- 6. pH Indicate for liquid or liquid portions of the waste. Check the appropriate boxes which cover the pH of the waste (water is 6-7). Use the "Range" space if appropriate. For solid or organic liquid wastes, indicate the pH of a 10% aqueous solution of the waste if applicable. Check "NA" for non-water soluble materials (e.g., bricks, dismantled tanks, empty drums, gases, rocks).
- 7. LIQUID FLASH POINT Indicate the liquid flash point obtained using the appropriate testing method (40 CFR 261.21). The liquid flash point is important for complying with DOT transportation requirements (49 CFR Part 173). Solids with flammable potential should be identified in Section G. OTHER CHARACTERISTICS OF THE WASTE, on the reverse side of the profile form.

PART E. TOTAL COMPOSITION OF WASTE

- 1. List all organic and/or inorganic components of the waste using specific chemical names. For each component, estimate the range (in percent) in which the component is present. In case of extreme pH (less than or equal to 2, or greater than or equal to 12.5), indicate specific acid or caustic species present. The total of the maximum values of the components must be greater than or equal to 100%, including water, earth, etc.
- 2. If this waste contains PCB, Cyanides, Sulfides, or Phenolics, indicate the concentration(s). If this waste does not contain these constituents, check the boxes under "Not Present". Indicate, by checking appropriate box, whether this was by Generator Knowledge or LAB Analysis.

PART F. METALS

Indicate whether metals content was determined by TCLP (Toxicity Characteristic Leaching Procedure), or redresents the total metals. For each metal, either check the box provided indicating that the metal content will not exceed the stated amount, or enter the actual metal content indicated by your test results. In the column below "ACTUAL", but the concentrations of each metal, if present, or "NA" if not present. If material has been tested, attach lab analysis, if not, check "Generator Knowledge" box.

PART G. OTHER CHARACTERISTICS OF THE WASTE

- 1. Indicate whether this wastestream (as a whole) is a "Wastewater" or "Nonwastewater". A Wastewater is defined as a waste containing less than 1% Total Suspended Solids (TSS), less than 1% Total Organic Carbon (TOC), and the rest of the waste is water. If the waste is not a Wastewater as defined above, then it is a Nonwastewater.
- 2. Indicate whether this wastestream is Organic, Inorganic, or Organo-metallic. Organic wastes are wastes which contain the element Garbon (e.g., solvents, petroleum products, wood, cloth). Inorganic wastes are wastes which contain no element of Carbon (e.g., water, glass, metallic wastes, masohry bricks). Organo-metallic wastes are wastes which are organic and have a Carbon-Metallic chemical bond (e.g., Tetraethy: Lead), or are Organic wastes which are commingled with Metallic wastes (e.g., Solvents contaminated with Chromium, rags contaminated with Lead, steel plates contaminated with an organic solvent or petroleum).
 - 3. Indicate whether the waste is any of the following:
 IGNITABLE SOLID A solid which will ignite and sustain compustion at temperatures below 140° F. (e.g., Sodium metal):
 WATER REACTIVE Reacts violently with water or when mixed with water forms toxic gases, vapors, or fumes in a quantity sufficient to present a danger to numan health or the environment (e.g., Magnesium metal, Annydrous Ammonia);
 REACTIVE (OTHER) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement, or if exposed to pH conditions ≤ 2 or ≥12.5 and it contains Sulfides or Gyanides and it can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health and the environment:
 OXIDIZER Yields oxygen readily to stimulate the compustion of organic matter, (e.g., Hydrogen peroxide);
 SHOCK SENSITIVE It is normally unstable and readily undergoes violent change without detonating (e.g., Organic ethers, Picric acid), IF YOU BELIEVE YOUR WASTE CONTAINS ANY MATERIAL THAT IS POTENTIALY SHOCK SENSITIVE, CONTACT YOUR CHEMPRO DIVISION SALES REPRESENTATIVE BEFORE SENDING A SAMPLE FOR ANALYSIS.

 If none of these properties apoly to the wastestream check the box "None Apply".

PART H. USEPA/STATE WASTE IDENTIFICATION

- HAZARDOUS/DANGEROUS WASTE Indicate if this waste is considered a Hazardous Waste as defined by RORA (40 CFR Part 261), or if it is a Washington State Dangerous waste as defined in WAC 173-303.
 DEPARTMENT OF ECOLOGY DESIGNATION The Washington Department of Ecology requires generators within the state of
- DEPARTMENT OF ECOLOGY DESIGNATION The Washington Department of Ecology requires generators within the state of Washington to designate whether their waste is classed as Dangerous Waste (DW) or as Extremely Hazardous Waste (EHW). Refer to WAC 173-303-090 thru 104 for the exact definitions for each waste type.
- 3. PCB REQULATED BY TSCA indicate whether the waste is or contains Polychlorinated biphenyl (PCB) in concentrations greater than or equal to 50 ppm, or is a waste contaminated with PCB from a source which originally contained PCB in a concentration greater than or equal to 50 ppm (e.g., contaminated soil from a leaking transformer containing PCB >50 ppm). PCB is regulated in concentrations greater than or equal to 50 ppm by the Toxic Substance Control Act (TSCA), which is administered by the USEPA.
- 4. List ALL waste codes or waste numbers which apply to this wastestream, including all EPA and state hazardous waste numbers or codes. NOTE: New regulations effective on May 8, 1990, require generators to list ALL characteristic waste numbers that are applicable to a waste, even if it is already designated by a listed waste number. For example, if you indicate that your waste is designated by the waste number F006 (Wastewater treatment sludges from plating baths), you must also indicate which metals are present in the waste by listing the applicable characteristic waste numbers) (e.g., D007 Chromium).

PART I. SHIPPING INFORMATION

- 1. Indicate if this waste is a USDCT Hazardous Materia: 49 CFR 172.101.
- 2. REPORTABLE QUANTITY Incidate what the Reportable Quantity (RQ is for this wastestream as determined by 49 CFR 172.101.
- Method of Shipment Indicate the anticipated method st of shipment by checking the appropriate boxies). If drums are to be used, see 49 CFR 173 for DOT drum specifications.
- 4. Amount to ship now/units Enter number of drums, gations, or other size containers ready to ship.
- 5. Anticipated Annual Volume/Units Enter the amount of this waste which will be generated and transported annually. Use appropriate units to describe this volume (e.g., public yards, gallons, pounds).

U.S. DOT DESCRIPTION - (FOR USE IN SECT. 11a-d OF UNIFORM HAZARDOUS WASTE MANIFEST).

- 6. PROPER SHIPPING NAME Oncose the proper DOT shipping name for this waste from the Hazardous Materials Table located in 49 CFR 172.101. If the amount of waste in any one container exceeds the Reportable Quantity, write the letters "RQ" IN FRONT OF the Proper Shipping Name. If the Proper Shipping Name for this wastestream is a generic shipping name containing the phrase "h.o.s." (e.g., Flammable Liquid, n.o.s.), you MUST indicate the phemical names of two predominant components in the waste which cause the waste to be hazardous, (e.g., Flammable Liquid, n.o.s.) (Acetone, Benzene). It is NOT appropriate to use waste numbers or appreciations in place of the chemical name. Not complying with the requirement to list the two constituents which most pregominantly contribute to the nazard of the waste filterial is a violation of DOT regulations.
 - NOTE: For STATE-ONLY regulated waste, it may not be appropriate to use a DOT Proper Shipping Name. If a state waste is not a USEPA nazardous waste and does not meet any DOT nazard class, it is recommended that you use the shipping name "Non-RCRA Waste Liduid or Solid". If a state-only waste does meet a DOT nazard class, the appropriate snipping name found in the Hazardous Materials Table (49 CFR 172.101, should be used.
- 7. HAZARD CLASS Enter the proper USDOT hazard class as determined by the Hazardous Materials Table found in 49 CFR 172.101.
- 8. DOTID NUMBER Enter the proper USDOT identification number located in the Hazardous Materials Table found in 49 CFR 172.101.
- 9. Enter the proper DOT Packing Group # located in the Hazarobus Materials Table toung in 40 CFR 172.101.
- 10. ADDITIONAL INFORMATION Enter ALL USEP4 Hazardous Waste Numbers (not state waste codes or numbers) which apply to this waste, other proper additional gescriptions as required by 49 GFR 172,203, or any other information which is not inconsistent with the waste material.

The blue-shaded area at the bottom of the profile sheet is for CHEMPRO DIVISION use only. Make no marks in this section.

PART J. SPECIAL HANDLING INFORMATION

Describe those hazards which you know or reasonably believe are or may be associated with short term or prolonged human exposure to this waste (29 CFR 1910.1200). If known, please identify any parcinogens present in this waste in excess of 0.1% (29 CFR 1910.1200 (d) (4)). Attach relevant documents as a part of your response if appropriate. If documents are attached, identify those attachments. If you have a current Material Safety Data Sheet, it may be attached. Failure to make an entry in PART J is considered to be a representation that you neither know nor believe that there are any adverse human health effects associated with exposure to this waste.

Also include in PART J any additional information that will aid in the management of the waste (e.g., protective clothing, transportation, treatment, storage, disposal).

PART K. GENERATOR CERTIFICATION

An authorized employee of the generator must sign and date this certification.

MAKE A COPY OF THIS GENERATOR'S WASTE MATERIAL PROFILE SHEET FOR YOUR RECORDS. SEND THE ORIGINAL AND ATTACHMENTS TO THE ADDRESS SHOWN ON THE FRONT OF THIS WORK SET OR TO THE ADDRESS PROVIDED BY YOUR CHEMPRO SALES REPRESENTATIVE.



GENERATOR'S WASTE MATERIAL PROFIL

PLEASE PRINT IN INK OR TYPE

E SHEET
CP# <u>56851</u>
NC=
Zip Code:
3. Phone: (=
Zip Code: #4X Phane # (=
☐ 'Waste Analysis
C Gravity: 5. Free Liquids: 3
≥ 12.5
Closed Cup Cpen Cup
icate if this waste contains any of the following ch :est method was used.
TOTAL GENERATOR KNOWLEDGE
ASTE No. LESS THAN ACTUAL D004
□ < 134 <u>cpm</u> ppm.

CHEMPRO DIVISION SALES REP.:				
A. GENERATOR INFORMATION				
1. Generator Name:		lenerator, USEPA 10 =		
3. Facility Accress:		Zip Code):	
4. Generator Contact	3. Title:	6. Phone: ()		
7. Consultant (if any): a. (Company:	9. Phone: ()		
8. MAIL INVOICES TO: 1. Generating Facility at acc				
2. Combany Name:		3. Phone: .		
4. Address:	State:	7'n Code	•	
5, Attention:		6. F4X Phone # (=		
C. WASTE INFORMATION				
1. Name Of Waste:				
2. Process Generating Waste (be specific:)	· · · · · · · · · · · · · · · · · · ·			
3. Generator has provided the following:	☐ Sample	☐ MSDS	☐ 'Waste Analysis	
D. PHYSICAL CHARACTERISTICS OF WASTE				
1. Color: 2. Physical State @ 70°F: Script Semi-Script Powder Other:		d	☐ Yes	
	1 6-8		lange:	
	· · · · · · · · · · · · · · · · · · ·			
E. TOTAL COMPOSITION OF WASTE 1. List all hazarcous and non-hazardous constituents of waste.	RANGE MIN MAX.	F. METALS: Indicate if this waste metals, and which test method v		
	·%	TCLP TOTAL	GENERATOR KNOWLEDGE	
	·%	METAL WASTE No. L	ESS THAN ACTUAL	
	o,] < 5 ppm ppm.	
		1	< 100 ppm ppm.	
	·] < 1 ppm ppm.	
	·%] < 5 ppmppm.	
	***	<u> </u>] < 5 ppm ppm.] < 0.2 ppm ppm.	
			< 1 ppm ppm.	
			< 5 ppm ppm.	
PLEASE NOTE: The TOTAL in the chemical composition must		Nicket (Ni)	l < 134 cpmppm.	
be greater than or equal to 100%.	TOTAL %	1	< 120 ppm ppm.	
Indicate if this waste contains any of the following: Not Present or Concentration		Zinc (Zn)	ppm.	
PCB	Lab Analysis	Copper (Cu)	ppm.	
Cyanidesppm	L Attached	Hexavalent Chrome	 :	
Phenolicsppm Sulfidesppm	Generator Knowledge			

G. OTHER CHARACTERISTICS OF THE WASTE		**************************************	
This waste is: (see instructions)	☐ Wastewater	☐ Nonwastewater	
2. This waste is: (see instructions)	Organic	Inorganic	Crgano-metailic
is this waste any of the following:	Ignitable Solid Oxidizer	Water Reactive Shock Sensitive	☐ Reactive (otner) ☐ None Apply
	No 2. Dept. of Ecology D No 4. List ALL Applicable	-	DW EHW
SHIPPING INFORMATION Is this a DOT Hazardous Material? Suis 2 guid Number of Units to Ship Now: 4. Number of Units to Ship Now: 4. Shipping Now: 4. S	s No 2. Reportab Bulk Solid 5. One Time - OR - Anticia	ole Quantity (RQ):	
US DOT DESCRIPTION: USE THE FUL	L BASIC DESCRIPTION	ON THE HAZARDOUS	WASTE MANIFEST
6. PROPER SHIPPING NAME		(d ģeneric "n.o.s.", iñģicatē ti	vo predominant constituents in parantnesi
7. HAZARD CLASS 8. DOT D NUMBER	9. PACKING GR	OUP NUMBER	0. ADDITIONAL DESCRIPTION
	that all information supmitted in t	nis and all attached documents	contains true and accurate descripti
K. GENERATOR CERTIFICATION: I hereb. berniy to of this waste material, and all relevant imprimation is.		nazargs in the possession of tr	
K. GENERATOR CERTIFICATION: I hereb. pertify to of this waste material, and all relevant information signature.		nazargs in the possession of tr	
K. GENERATOR CERTIFICATION: I hereb. berdiy to of this waste material, and all relevant information. I. SIGNATURE		nazargs in the possession of tr	
C. GENERATOR CERTIFICATION: I herep, pertify to of this waste material, and all relevant information signature. SIGNATURE NAME type or or fit. FOR CHEMPRO DIVISION USE ONLY	regarding known or suspected	nazargs in the possession of tr 2. TITLE 4. DATE ADDITIONAL	ne ģeneratōr has beēn disclosed.
FOR CHEMPRO DIVISION USE ONLY Analyze sample on the following basis:	regarding known or suspected Total TCLP	ADDITIONAL	ne çeneratör has been disclosed. TESTING REQUIREMENTS
K. GENERATOR CERTIFICATION: I hereb. certify to of this waste material, and all relevant information. SIGNATURE NAME type or print: FOR CHEMPRO DIVISION USE ONLY Analyze sample on the following basis: METALS: Arsenic (As) Barium (Ba) Cadmium (Cd) Cadmium (Cd) Chromium (Cd)	regarding known or suspected Total TCLP	ADDITIONAL SP TOX	TESTING REQUIREMENTS
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K. GENERATOR CERTIFICATION: I hereb. permity to of this waste material, and all relevant information is signature. SIGNATURE B. NAME type or print: FOR CHEMPRO DIVISION USE DNLY Analyze sample on the following basis: METALS: Arsenic (As) Selenium (S) Sarium (Ba) Silver (Ag) Selenium (Cd) Nickel (Nichel (Total TCLP Sel DISCHARGE METALS: TO METALS (D004-11): F006 TCLP METALS: OTHER (list separately):	ADDITIONAL DATE ADDITIONAL EP TOX (Cd. Cr. C) (As. Ba. C) (As. Ba. C)	TËSTING REQUIREMENTS Ou. Pb. Ni. Zn) Od. Cr. Pb. Hg, Se. Ag) Pb. Ni. Ag) Od. Cr. Hg. Pb. Se. Ag. Cu, Ni. Zn, T
CYANIDS	Total TCLP Sel DISCHARGE METALS: TC METALS (D004-11): F006 TCLP METALS: OTHER (list separately): BETX ppm OTHER	ADDITIONAL DATE ADDITIONAL EP TOX (Cd. Cr. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (As. Ba. (Cd. Cr. F. (Cd. Cr. Cr. F. (Cd. Cr. F. (Cd. Cr. Cr. Cr. Cr. Cr. Cr. Cr. Cr. Cr. Cr	TËSTING REQUIREMENTS Ou. Pb. Ni. Zn) Od. Cr. Pb. Hg, Se. Ag) Od. Cr. Hg. Pb. Se. Ag. Cu, Ni. Zn, T
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GENERATOR'S CHAIN OF CUSTODY RECORD

1. SAMPLING METHOD

Questions concerning sample, walvér šnould be reférred to your CHEMPRO DIVISION Sáles Réprésentative.

Write in the sampling method employed.

This sample should be collected in accordance with "Test Methods for the Evaluation of Solid Waste, Physical Chemical Methods", SW846, USEPA, Office of Solid Waste. Washington D.C. 20460 and/or 40 CFR 261-Appendix I. A suitable samble container for most wastes is a wide mouth glass cottle with a plastic screw-on lid having a non-reactive liner. Plastic containers are recommended for strong caustics or flyorides. Fil to approximately 90% of capacity to allow for expansion during transportation. The beel-off label on this form must be completed prior to removal from the form. The label must be attached to the sample container, not to the shipping carton.

If this waste is a hazardous material, the sample must be backaged and shipped in accordance with USDOT regulations (49 CFR 171.2) and any specific requirements imposed by the carrier, improperly packaged samples may be disposed of upon receipt.

2. SAMPLE SCURCE

The sampler is to describe exactly from where the sample was taken (e.g. conveyor, drum, fagodh, bloe, pit, bond, tank, vat).

3. PROFILE APPROVAL FEE

Indicate by checking appropriate box if you are enclosing your profile approved fee or if you have established credit with Chempro Division and wish to be billed. If your company requires a P.O. #, write that number here.

4. SAMPLE LABEL

THE SAMPLE LABEL MUST BE COMPLETED BEFORE IT IS REMOVED FROM THIS FORM.

Apply the completed peel-off label to the container which actually holds the sample - not to the shipping carton.

1. DATE SAMPLED

- Enter the month, day, year.

2. PROFILE NUMBER

- Enter the black number.

3. GENERATOR

. Enter name of generating facility.

4. WASTE DESCRIPTION

- Enter a name which is generally descriptive of the waste (e.g., paint sludge, still bottoms, PCB oil, mixed acids, cyanide plating waste) as it appears on the profile sheet sect. C-1.

5. ANALYZE FOR:

· "Profile check" is preprinted.

6. PRINT SAMPLER'S NAME - Enter the name of the person who actually took this sample.

7. SAMPLER'S SIGNATURE

- The sampler must sign in the space provided.

Remove the completed peel-off label and affix it to the sample container at the time of sampling. If this label is lost or destroyed, the sample must be laceled with equivalent information, including the Waste Profile Number. If the Cartification of Representative Sample Form is lost or destroyed, please contact your CHEMPSO DIVISION sales représentative to obtain a new ône.

5. CHAIN OF CUSTODY

Each person who handles the sample must sign when the sample passes from one individual to another.

RELINQUISHED BY - Person who is relinquishing the sample signs and gives the date and time.

RECEIVED BY - Person who receives the sample signs and gives date and time.

- PLEASE TURN PAGE TO COMPLETE LABEL -

GENERATOR'S CHAIN OF CUSTODY RECORD

CP# - 56851
WASTE PROFUE SHEET NUMBER This completed form must be returned, with the representative sample to: BURLINGTON ENVIRONMENTAL - CHEMPRO DIVISION 2203 AIRPORT WAY SO. . SUITE 400 SEATTLE, WASHINGTON 98134 (206) 243-6776 INSTRUCTIONS Collect a representative 1 pint sample of the waste described in the above referenced CHEMPRO DIVISION Profile using a glass jar, with a screw-on lid. If the sample is oil, we will need a 1 quart sample. A representative sample is one obtained using any of the applicable sampling methods cited in 40 CFR 261-APPENDIX 1. Fill in the sampling information in the spaces provided below. Comblete all items on the label, except the Laboratory ≠, then remove the label and affix it to the sample bottle. If you have problems obtaining a representative sample of your waste, please contact your ICHEMPRO sales representative. SAMPLING METHOD Indicate method utensils employed in sampling: SAMPLE SOURCE (e.g., drum, tank, bit) PLEASE BILL P.O. # PAYMENT ENCLOSED 3. PROFILE REVIEW FEE: 4. SAMPLE LABEL - COMPLETE LABEL BEFORE REMOVING Date Sampled: _____ CP #: ____ 56851 Generator: _ Waste Description: (USE BALL POINT PEN ON LABEL Analyze for: Profile Check PRESS FIRMLY) Sampled by: _ Signature: aboratory (# For lab use only Affix the peel-off label to the sample and ship it and this form to the address noted above. 5. CHAIN OF CUSTODY Each person who handles the sample must sign below when the sample passes from one to another. Time Time Relinquished by: (Signature) Date Received by: (Signature) Date

APPENDIX D

Standard Operating Procedures for the pH Meter

STANDARD OPERATING PROCEDURES FOR THE pH METER

1 INTRODUCTION

The purpose of this Standard Operating Procedures (SOP) Manual is to inform field personnel of the theory, operation, quality control, maintenance, and documentation of the pH meter. The pH meter logbooks must be obtained from and returned to the environmental chemist. This enables the chemist to perform routine maintenance and calibration of the instrument.

1.1 QUICK REFERENCE OPERATION

- 1. Turn instrument on, electrode to meter.
- 2. Set MODE switch to pH.
- 3. Calibrate by using two buffer solutions and rinse electrode twice before immersing in sample.
- 4. Measure temperature of sample.
- 5. Set temperature adjustment to sample temperature.
- 6. Place electrode in sample and allow to stabilize for 45-60 seconds.
- 7. Record results.
- 8. Rinse twice with distilled water and place electrode in distilled water between measurements.

2 THEORY OF OPERATION

The pH of a solution may be defined as the negative log of the hydrogen ion concentration:

$$pH = log \ \underline{1} = -log \ [H^+],$$

where [H⁺] is the hydrogen ion concentration in moles per liter. For a neutral solution:

$$[H^+] = 1.0 \times 10^{-7}$$

 $pH = -log (10^{-7}) = -(-7)$
 $pH = 7$

It should be kept in mind that pH relates to a power of 10. Hence, a solution of pH = 1 has a hydronium ion concentration 100 times that of a solution of pH = 3 (not three times). Furthermore, since the pH is related to a negative exponent, the lower the pH value, the larger the concentration of hydronium ion. At pH = 7, a solution is neutral. Solutions with pH below 7 are acidic; those with pH above 7 are alkaline.

A pH meter employs potentiometric methods for pH determination. Since the electromotive force of a cell is dependent upon activities and not concentrations, we refine the definition of pH as the negative logarithm of the activity of the hydronium ion concentration.

One electrode is a hydrogen electrode in which the electrolyte is the solution being studied. This electrode is connected by means of a salt bridge to a normal calomel electrode. The calomel electrode utilizes as the electrolyte a normal solution of potassium chloride which is saturated with mercurous chloride. The electrode proper consists of mercury metal at the bottom of the tube, overlaid with a paste of mercurous chloride (calomel) and mercury.

There is an indeterminable liquid-junction potential at the salt bridge. The dependence of the electromotive force of the cell upon the activities of the species present may be expressed by adapting the Nernst equation.

$$E = E^{0} - 0.05916 \log [aH^{+}]^{2} [aCl^{-}]^{2} + E$$

$$2 [aH_{2}]$$

$$E = E' - 0.5916 \log [aH^{+}]$$

For the normal calomel electrode in air at 25° C, E' is 0.2825 V. Therefore,

$$E = 0.2825 + 0.05916 \text{ (pH)}$$

$$pH = \underbrace{E - 0.2825}_{0.05916}$$

This expression may be regarded as an operational definition of pH.

In place of the hydrogen electrode, a glass electrode is usually employed for the determination of pH. A glass electrode consists of a reference electrode containing a solution of known pH sealed in a membrane of special glass. In the determination of the pH of a solution, the glass electrode and calomel electrode are immersed in the solution being studied. This is made into a single electrode called a combination electrode. The combination electrode is the electrode used out in the field.

The electromotive force of the complete cell depends upon the difference in potential across the glass membrane that separates solutions of different pH and is found to respond to changes in pH in the same way that an assembly using a hydrogen electrode does. The value of E' for a cell using a glass electrode depends upon the electrode used in the construction of the combination electrode as well as the other reference electrode employed in the complete cell. pH meters employ glass electrodes and are usually calibrated in pH units rather than volts.

3 **DETAILED FIELD PROCEDURES**

3.1 INSTRUMENT CHECK-OUT PROCEDURES

- 1. Turn power switch to ON position.
- 2. Turn MODE switch to pH.
- 3. Attach BNC short plug to BNC connector and adjust calibration knob to read a steady 7.00.
- 4. Remove short plug and attach electrode to meter.

3.2 CALIBRATION FOR FIELD AND LABORATORY

- 1. Calibrate meter at beginning of each day. Check calibration in the middle of the day, and at end of day or after last measurement when in use.
- 2. Record in logbook the buffers used for calibration and their stock numbers, time, and initials of person performing each calibration.
- 3. Use two buffer standard solutions for maximum accuracy, either pH 4 and 7, or 7 and 10.
- 4. Sample and buffer temperatures must be equal.
- 5. Set temp/slope control to buffer temperature, 0° C.
- 6. Rinse electrode twice with deionized water and place in pH 7.0 buffer solution and stir moderately.
- 7. Set MODE switch to read pH. Allow reading to stabilize (30-45 seconds), then gently adjust calibration control so that 7.00 is displayed (any value from 6.98 to 7.02 is acceptable).
- 8. Remove electrode from buffer solution and rinse twice with distilled water.
- 9. Place electrode in second buffer solution (pH 4.00 or pH 10.00). Stir moderately and allow reading to stabilize (30-45 seconds).

- 10. Adjust temperature slope control until correct pH value of second buffer is displayed. Remove electrode and rinse it twice with distilled water.
- 11. The readings should differ from the actual standard pH value by 0.02 or less. If this is not the case, recalibrate with pH 4 and 7, or 7 and 10, standard buffer solutions.

3.3 SAMPLE MEASUREMENT, FIELD

- 1. Rinse probe twice with deionized water.
- 2. Place electrode in sample and stir moderately. Allow reading to stabilize (45-60 seconds).
- 3. Record the date, time, sample number and pH in logbook.
- 4. Remove electrode and rinse twice with distilled water.
- 5. Repeat steps 1-3 for each sample.
- 6. Electrode can be placed in distilled water for short periods of time between measurements but must be rinsed thoroughly with distilled water first to prevent cross-contamination of samples.
- If a sample temperature differs significantly from the buffer temperature used to calibrate the instrument, an adjustment can be made. Raise or lower temp/slope control from its current setting by the difference between the actual buffer temperature and the sample temperature. Record this adjustment in logbook, and time adjustment was made.

4 QUALITY CONTROL / QUALITY ASSURANCE (QA/QC)

4.1 FIELD

- 1. When making pH measurements, use duplicate measurements to assure accuracy of measurement. To do this after obtaining the first reading, remove probe, rinse twice with distilled water and re-insert into same sample. Let reading stabilize and compare readings. The two readings should differ by 0.02 or less. Record results. If in error, repeat procedure a third time. If error persists, troubleshoot problem, and/or recalibrate instrument. Record all actions in logbook along with all readings.
- 2. If erroneous readings are suspected, take several readings of a known pH standard and recalibrate. Compare and log in book.
- 3. Document all problems with instrument and probe in logbook and notify project supervisor or call environmental chemist.
- 4. Take readings only in sample temperatures between 0°C and 80°C.
- 5. Check pH standards by taking reading from two separate previously calibrated pH meters and comparing results. This should be done at least once a month.

5 MAINTENANCE

5.1 FIELD MAINTENANCE

Air bubbles may appear in the electrode during transit. To remove, hold the probe vertically and gently tap sides to release trapped air. If instrument is in field for an extended time, the operator needs to check the electrode solution. When low, add filling solution per directions under laboratory maintenance in this manual. Record date and initials in appropriate places if electrode solution is added.

5.2 LABORATORY MAINTENANCE

Check the electrode filling solution after each field use. The electrode solution will discharge into the sample and will run low, damaging the electrode. When 2/3 full, add appropriate solution by sliding rubber ring down past fill hole. Then pour solution into fill hole until full. Replace ring over fill hole. Tap sides gently to loosen air bubbles while holding the electrode vertical. Recalibrate using standards after letting electrodes stabilize (approximately 15 minutes).

Check battery in instrument once a month and replace as needed. Check the battery if you suspect erroneous readings and when the machine will not calibrate properly.

6 STORAGE

Disconnect electrode connector, replace short plug and turn machine off.

6.1 FIELD

Place a few drops of water in rubber cap and place over the electrode to increase the life of the probe when not in use.

6.2 LABORATORY

For periods of long storage, remove batteries to prevent leakage and corrosion to the instrument. Place in storage case and keep in cool, dry place.

6.3 **CLEANING - LABORATORY**

Soak electrode in 0.1M HCl or HN0₃ for 15 minutes, followed by soaking in pH 7 buffer for 30 minutes.

Removal of deposits:

- 1. Protein: digest with 1% pepsin in 0.1M HCl.
- 2. Inorganic: rinse with 0.1M tetrasodium EDTA solution.
- 3. Grease and oil: rinse with mild detergent or methanol solution.
- 4. After each of these methods, soak the electrode in pH 7 buffer for 30 minutes.

ORION
Laboratory Products Group
Orion Research Incorporated

MODEL 210 INSTRUCTION MANUAL

INSTRUCTION SHEET gel-filled combination pH electrode models 91-05, 91-06, 91-07, 91-08 91-15, 91-16 - 91-25, 91-26 91-35, 91-36

introduction

The ORION gel-filled combination electrode is designed for routine pH measurements under rugged conditions. The unbreakable body extends beyond the pH sensing glass bulb for protection, allowing the electrode to be used as a stirring rod. The sealed reference section, permanently filled with a KCl gel, never needs refilling. The electrodes are:

- conventional electrode (91-05, 91-06, 91-07, 91-08) for general purpose pH measurement.
- semi-micro electrode (91-15, 91-16) for pH measurement in test tubes. Provided with each electrode is an adapter which allows the use of the ORION universal electrode holder (cat. no. 910002).
- flask electrode (91-25, 91-26) for pH measurement in a tall-necked flask.
- flat-surface electrode (91-35, 91-36) for pH measurement on solids or semi-solid substances.

required equipment and solutions

buffers-two buffers: one near pH 7; and, one near sample pH.

pH meter—any ORION or other meter with appropriate input jacks can be used. The following connector configurations are available:

- 91-05, 91-15, 91-25, and 91-35 have standard U.S. Connectors.
- 91-06, 91-16, 91-26, and 91-36 have a BNC connector for use with hand-held digital pH meters.
- 91-07 has a miniature phone plug for use with the Corning model 5 and 610A pH meters.
- 91-08 has a DIN connector.

preliminary to operation

- 1. The electrode tip is covered by a cap which protects the electrode and keeps it from drying. Pull the cap off and save for storage. There is normally no need to soak the electrode before use as the cap contains soaking solution.
- 2. If bubbles are seen in the bulb area of the electrode, shake the electrode downward. This action will help eliminate bubbles that may have been generated during shipment.
- 3. Connect electrode to meter.

single-buffer calibration

This procedure is for routine measurements. Electrode slope should be checked periodically with a two-buffer calibration.

- 1. Choose a buffer which is near the expected sample pH.
- 2. Buffer should be at room temperature. If samples are at varying temperatures, temperature compensation is recommended. (See meter instruction manual.)
- 3. Turn the meter slope indicator dial to 100% and the temperature knob to the temperature of the buffer. (Some meters may use other controls for this procedure.)
- 4. Rinse electrode with distilled water and place in the buffer.
- Wait for a stable display. Using the calibration control, set the meter to the pH value of the buffer at its measured temperature. (A table of pH values at various temperatures is supplied with the buffer.)
- 6. Rinse electrode with distilled water and place in sample.

CHOCHERCE COMMETCE

This procedure is recommended for precise measurement.

- 1. Choose two buffers which bracket the expected sample pH. The first should be near pH 7 and the second near pH 4 or pH 10.
- 2. Insure that buffers are at room temperature. If samples are at varying temperatures, temperature compensation is recommended. (See meter instruction manual.)
- 3. Rinse electrode with distilled water and place in pH 7 buffer.
- 4. Wait for a stable display. Using the calibration control, set the meter to the pH value of the buffer at its measured temperature. (A table of pH values at various temperatures is supplied with the buffer.)
- 5. Rinse electrode with distilled water and place in second buffer.
- 6. When display is stable, set meter to the actual pH value of the buffer using the temperature compensation or slope control. (Some meters may use other controls.)
- 7. Rinse electrode with distilled water and place in sample.
- 8. When display is stable, record pH.

measuring hints

- Shake off drops of solution to prevent carryover of one solution to another.
- Keep buffers and samples at approximately the same temperature. Compensate for small temperature variations with the temperature knob.
- Check electrode operation periodically with a two-buffer standardization. If readings in the buffer drift or if slope is below 92% (in meters with no percent slope control, temperature control reading is less than 5°C), follow cleaning procedure.
- Between measurements leave the electrode in the open-air laboratory environment, not in distilled water.

cleaning procedure

general—soak electrode in 0.1 M HCl or HNO₃ for 15 minutes, followed by soaking in pH 7 buffer for 30 minutes.

removal of deposits

protein-digest with 1% pepsin in 0.1 M HCI*.

inorganic-rinse with 0.1 M tetrasodium EDTA solution.*

grease and oil-rinse with mild detergent or methanol solution.*

* After any of these cleaning procedures, soak the electrode in pH 7 buffer for 30 minutes.

electrode storage

When not in use, cover the electrode tip with the protective cap used for shipment.

specifications

electrode	ρН	temp. range	isopotentia point	i length (cap included)	body diameter	cap diameter	cable length
91-05, -06,	•					·	
-07, -08	0 - 14	$0 = 80^{\circ}C$	pH 7	140 mm	12 mm	16 mm	100 cm
91-15, -16	0 - 14	0 - 80°C	pH 7	180 mm	6 mm	10 mm	100 cm
91-25, -26	0 - 14	0 - 80°C	pH 7	335 mm	8 mm	16 mm	100 cm
91-35, -36	0 - 14	0 - 80°C	pH 7	140 mm -	12 mm	16 mm	100 cm

SPECIAL INSTRUCTION FOR USING YSI 3311X SCT PROBES

When using YSI 3311X SCT Probes with cable lengths greater than 50 feet, a very small but constant error in all conductivity readings will be introduced by the extra cable length. This error may be determined as follows by slightly altering the standard operating procedure when setting up the YSI Model 33 SCT Meter to make conductivity measurements.

SETUP:

- A. Adjust meter to zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity (umhos/cm) scale.
- B. Calibrate the meter by turning the switch to redline and adjusting the meter needle with the redline control to the red line on the scale. If this cannot be accomplished, replace the batteries.
- C. Plug the dry probe into the probe jack on the side of the instrument. Any difference between the reading with and without the dry probe plugged into the instrument represents the error introduced by the extra cable length. This error will generally be insignificant on the X10 and X100 conductivity scales. The error has no effect on temperature and salinity readings.
- D. Put the probe in the solution to be measured and follow the standard procedures shown in the YSI Model 33 instruction manual.

ADDENDUM; SA 230 INSTRUCTION MANUAL

On page 5 under **Single Buffer Standardization** (With ATC) steps 1 and 2 should read as follows:

- Plug ATC probe into input jacks and adjust °C/slope control knob to 25°C. Slide mode switch to temperature. Verify that ambient temperature is displayed.
- 2. If incorrect, adjust °C/slope control knob until actual temperature is displayed.

OSION

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ADDENDUM, SA 210 AND SA 230 INSTRUCTION MANUALS

If the line converters that ORION supplies, Cat. No. 020121 and 020120, are not available, any line converter meeting the following specifications may be used.

Converter for 120 VAC to 9 VDC

This specification describes an AC-to-DC power supply for use with ORION Products.

Electrical Specifications

- 1. The power supply shall furnish rectified, filtered, unregulated DC voltage.
- 2. The input voltage shall be 100-130 VAC, 47-63 Hz.
- The open circuit output voltage shall not exceed 15.5 VDC at an input voltage of 130 VAC, 60 Hz.
- The unit shall produce an output voltage not less than 9.0 VDC with a load of 200 MADC at an input voltage of 115 VAC, 60 Hz.

Mechanical Specifications

- The power supply shall plug into two blade wall outlets that are standard in North America for 115 VAC service.
- Output cord shall terminate in a standard 3.5 mm diameter phone plug. The tip shall be negative, the sleeve positive.

Safety

 The power supply shall be UL listed and CSA approved.

Converter for 220 VAC to 9 VDC

This specification describes an AC-to-DC power supply for use with ORION Products.

Electrical Specifications

- The power supply shall furnish rectified, filtered, unregulated DC voltage.
- 2. The input voltage shall be 200-240 VAC, 47-63 Hz.
- The open circuit output voltage shall not exceed 15.5 VDC at an input voltage of 240 VAC, 50 Hz.
- The unit shall produce an output voltage not less than 9.0 VDC with a load of 200 MADC at an input voltage of 220 VAC, 50 Hz.

Mechanical Specifications

- The power supply shall plug into wall outlets that are appropriate for the area.
- 2. Suggested cord length is 1.5 meters long.
- 3. Output cord shall terminate in a standard 3.5 mm diameter phone plug. The tip shall be negative, the sleeve positive.

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GENERAL INFORMATION

Introduction

The ORION Model SA 210 is a portable battery-operated digital pH meter for field, plant or laboratory use. It is designed for versatile, easy operation, and can be used in or out of the accompanying carrying case. The instrument is lightweight and designed to fit comfortably into the hand. All controls are on the meter face which affords one hand calibration.

The instrument measuring range is –1999 to + 1999 mV, and pH is displayed to two decimal places. The meter has a large easy to read LCD display.

Instrument Description See Figure 1.

- 1 **On/Off switch**: Slide switch controls power to meter.
- 2 LCD display: Model SA 210 automatically displays data in large numerals with negative polarity sign and decimal point. pH values are displayed from 0 to 14 with 0.01 pH unit resolution. Millivolt range is -1999 to + 1999. Dissolved oxygen is measured from 0 to 14 ppm when meter is used with 97-08 Oxygen Probe.
- 3 **Mode Control**: Provides operator with choice of measuring sample in either pH or mV mode.
- 4 Calibration Control: Used to standardize the meter/electrode system in buffers of known pH.
- 5 Temperature/slope Control: Compensates for variation in electrode slope or solution temperature.
- 6 Electrode Connection: Accepts BNC connector from combination electrodes and pin tip jack available for use with separate half-cell reference electrodes.

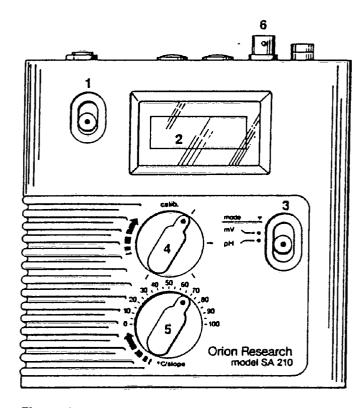


Figure 1 Face of the SA 210

INSTRUMENT SET-UP See Figure 2.

Support Rod

- 1. Attach support rod base to side of meter carrying case and tighten clamp screw.
- 2. Insert support rod into base. Tighten by turning rod clockwise.
- 3. Attach electrode holder to top of support rod.

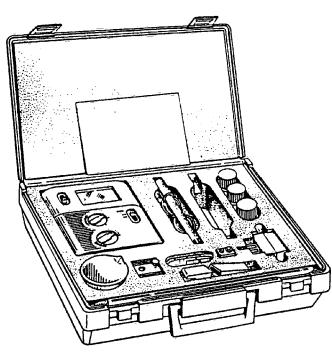


Figure 2 Model SA 210 and accessories

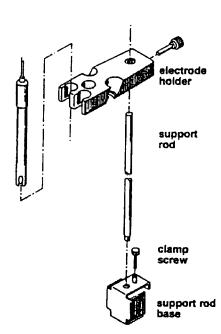


Figure 3
Support rod and clamp

Power Source

ORION Model SA 210 operates on one non-rechargeable 9-volt alkaline battery. Optional AC line adapters are available for both 110 and 220 volt mains. Refer to ACCESSORIES, page 7.

Battery Installation See Figure 4.

- Remove access panel on back of meter. No tools are required, simply slide cover towards bottom of meter.
- Attach battery connector clip of meter to battery terminals, install battery and replace access panel.

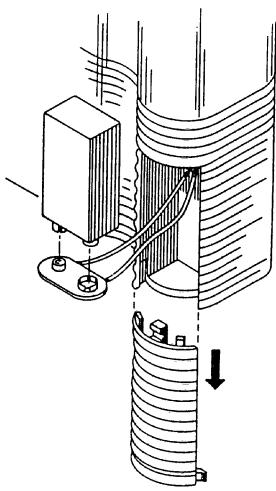


Figure 4
Rear access panel removed

Electrode Connections

- 1. Attach electrodes with BNC connectors directly to the top of the meter. Refer to **Figure 5**.
- Electrodes without BNC connectors may be used with a commercially available BNC adapter (ORION Cat. No. 090033). See Figure 6.

If using a combination electrode, electrode connection 3 is not used.

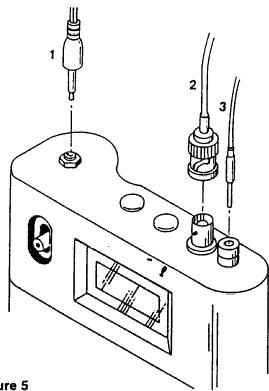


Figure 5
Electrode connections

Legend

- 1 AC line adapter
- 2 BNC connector
- 3 reference pin-tip connector

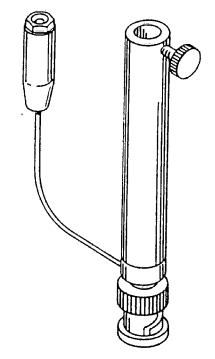


Figure 6 U.S. Standard connector to BNC adapter

Meter Check-Out Procedure

- 1. Slide power switch to ON position.
- 2. If using optional AC line adapter, connect it to meter and appropriate power source. Proceed to step 4.
- 3. Slide mode switch to pH. If LO BATT indicator on LCD remains on, battery must be replaced.
- Attach BNC shorting plug (ORION Cat. No. 090045) to BNC connector on top of meter. Slide mode switch to pH. Adjust calib knob to read a steady 7.00. If this cannot be done, refer to TROUBLE-SHOOTING, page 6.
- 5. Remove the shorting plug. After successful completion of steps 1-4, the meter is ready for use with an electrode.

MEASUREMENT PROCEDURES

pH Measurements

Single Buffer Standardization

- Şample and buffer temperature must be equal. Set temp/slope control to buffer temperature (°C).
- Place electrode in a buffer solution with pH value within 1.5 units of the expected sample value and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control so that correct buffer value at that temperature is displayed.
- Remove electrodes from the buffer solution and rinse.
- Place electrodes into sample and stir moderately.
 Allow reading to stabilize.
- 6. Record pH value displayed.

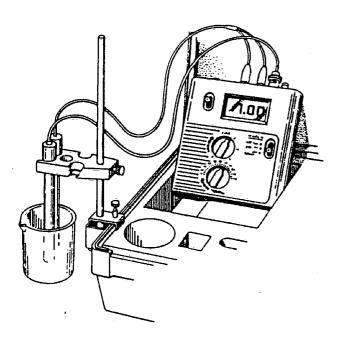


Figure 7 SA 210 meter set up for sample measurement

Two Buffer Standardization

NOTE: For maximum accuracy, perform a two buffer calibration once at the beginning of each day. This procedure provides the correct setting for temp/slope control. Subsequent measurements are made after a single buffer calibration.

- 1. Sample and buffer temperature must be equal. Set temp/slope control to buffer temperature (°C).
- 2. Place electrode in pH 7 buffer and stir moderately.
- Slide mode switch to pH. Allow reading to stabilize, then adjust calib control so that correct buffer value at that temperature is displayed.
- 4. Remove electrode from the buffer solution and rinse.
- 5. Place electrode in second buffer and stir moderately. Allow reading to stabilize.
- Adjust temp/slope control until correct value of second buffer is displayed. Remove electrode from solution and rinse.
- Place electrode in sample and stir moderately. Allow reading to stabilize.
- 8. Record pH value displayed.

NOTE: If sample temperature differs significantly from the buffer temperatures used to calibrate, an adjustment can be made. Raise or lower the temp/slope control from its current setting, described in step 6, by the difference between the actual temperature and the sample temperature.

Potentiometric Measurements

Potentiometric titrations are performed in mV mode using either ion selective or redox electrodes with BNC connectors. Detailed instructions for any ORION electrode are given in the electrode instruction manual. Titration instructions are included in ORION Redox Electrode (Model 96-78) Instruction Manual, or in standard analytical chemistry texts. U.S. Standard to BNC adapters are available from ORION (Cat.No. 090033).

Dissolved Oxygen Measurement

Dissolved oxygen measurements are displayed in ppm when ORION Model 97-08-99 Dissolved Oxygen Electrode is used with Model SA 210. Follow the instructions in the electrode manual. Be sure to set meter mode switch to pH.

TROUBLESHOOTING GUIDE

The following section covers troubleshooting that can be performed without special tools or skills. The ORION Technical Service Personnel can be consulted for troubleshooting advice by calling 1-800-225-1480 or 617-864-5400. Outside North America contact your local authorized ORION Representative.

Malfunction	Possible Cause	Remedy		
No Display	No power to meter	Check that switch is in ON position.		
		Replace battery.		
		Check that adapter is receiving power and is plugged in securely.		
More than one decimal displayed	mode switch is between positions	Adjust switch to proper position.		
Erratic readings or drift. Readings out of range.	Electrode failure	Follow meter checkout procedure. If meter okay, check electrode.		

INSTRUMENT WARRANTY

ORION RESEARCH INCORPORATED warrants this instrument will operate for one year from the date of purchase when used under normal laboratory conditions, and in accordance with the operating limitations and maintenance procedures given in the instruction manual. In the event of failure within the warrant period, ORION, or its Authorized Dealer, will, at ORION'S option, repair or replace the non-conforming instrument at no charge to the customer.

THE WARRANTY DESCRIBED ABOVE IS EXCLUSIVE AND IN LIEU OF ANY OTHER WARRANTY. WHETHER STATUTORY, EXPRESS OR IMPLIED, IN-CLUDING BUT NOT LIMITED TO, ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE AND ALL WARRAN-TIES ARISING FROM COURSE OF DEALING OR USAGE OF TRADE, EXCEPT TITLE. THE BUYER'S SOLE AND EXCLUSIVE REMEDY IS FOR REPAIR, OR REPLACEMENT OF THE DEFECTIVE INSTRUMENT OR PART, OR REFUND OF THE PURCHASE PRICE: BUT IN NO EVENT SHALL ORION (ITS CONTRAC-TORS AND SUPPLIERS OF ANY TIER) BE LIABLE TO THE BUYER OR ANY PERSON, IN CONTRACT OR IN TORT (INCLUDING NEGLIGENCE) FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSE-QUENTIAL DAMAGES.

Representations and warranties made by any person, including dealers, representatives and employees of DRION, which are inconsistent or in conflict with the terms of this warranty shall not be binding upon ORION unless in writing and signed by one of its officers.

REPAIR AND SERVICE

A Return Authorization Number must be obtained from DRION Laboratory Products Customer Service before aturning any product for in-warranty repair, replacement or credit. Contact ORION by calling 800-225-1480 (USA outside Mass.) or 617-864-5400. Dutside the USA and Canada consult your local incountry, authorized ORION sales agent/distributor for product service information.

OPTIONAL ACCESSORIES

Description

Cat. No.

Cat. No.	Description
815600	Ross®epoxy body, bulb guard combination pH electrode
9104BN	Laboratory grade combination pH electrode (BNC connector)
910600	GX-series epoxy body, gel-filled combination pH electrode (BNC connector)
912600	GX-series epoxy body, gel-filled flask combination pH electrode (BNC connector)
913600	GX-series epoxy body, gel-filled flask combination pH electrode (BNC connector)
915600	RX-series refillable, epoxy body combination pH electrode (BNC connector)
9162BN	Combination pH electrode with rugged bulb (BNC connector)
9163BN	Combination pH electrode with needle shape (BNC connector)
910004	pH 4 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910007	pH 7 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910009	pH 9 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910104	pH 4.01 buffer, 475 ml bottle
910107	pH 7.00 buffer, 475 ml bottle
910110	pH 10.01 buffer, 475 ml bottle
970899	Dissolved oxygen electrode
910002	Electrode holder
090033	U.S. Standard to BNC connector adapter
090045	Shorting plug
020120	110V AC line adapter
020121	220V AC line adapter
020041	Shoulder strap and meter holder for hands free operation
020042	Carrying case with foam insert, without meter or accessories
020043	Support rod and guide
020044	Accessory pack includes two 60 ml bot- tles and one 150 ml beaker
020045	Electrode rod stand for bench-top meter use outside carrying case

SPECIFICATIONS

Model SA 210, ORION Cat. No. 021000 portable pH/mV meter for hand held and bench top use. Digital LCD meter comes in carrying case with combination pH electrode and accessories, ready for immediate use. 110 and 220 volt adapter(s) available for AC line use.

Modes

pH, mV, O₂ (with O₂ probe)

pH range

pH 0 to 14

pH resolution

0.01

mV range

-1999 to +1999 mV showing negative polarity sign

mV resolution

1 mV

Temperature compensation

Manual

Sample temperature range

-5 to 105°C

Input impedance

> 100,000 megohms

Instrument drift

<50 microvolts/°C

Input blas current

 $< \pm 1$ pico amp at 25°C and $< \pm 4$ pico amps over full operating range

Environmental requirements

5 to 45°C and 5 to 80% relative humidity, non-condensing

Isopotential point

pH 7 (fixed)

Power requirement

One 9 volt battery. Optional 110 or 220 volt line adapter(s) available for AC line use.

Inputs

BNC combination and separate pin tip reference jack

Meter dimensions

14 cm X 14 cm X 4 cm

Meter weight

0.5 kg

Meter case

Splash-proof, chemical resistant

Carrying case dimensions

38.1 cm x 27.9 cm x 11.4 cm

Carrying case weight

1.8 kg

NOTICE OF COMPLIANCE

This meter may generate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type-tested and found to comply with the limits for a Class B computing device in accordance with specifications in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If the meter does cause interference to radio or television reception, which can be determined by turning the unit off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna.
- Relocate the meter with respect to the receiver
- Move the meter away from the receiver.
- Plug the meter into a different outlet so that the meter and receiver are on different branch circuits.

If necessary, the user should consult the manufacturer or an experienced radio/television technician for additional suggestions. The user may find the following booklet prepared by the Federal Communications Commission helpful:

"How to Identify and Resolve Radio-TV Interference Problems."

This booklet is available from the U.S. Government Printing Office, Washington, D.C. 20402, Stock No. 004-000-00345-4.

ADDENDUM

SA 210/230 INSTRUCTION MANUALS

To further clarify the use of the ORION Model 97-08 Dissolved Oxygen Electrode (Cat. No. 970899 with BNC connector) with your new meter the following instructions are included:

- 1. Connect electrode to the meter.
- 2. With the electrode mode switch in the OFF position, switch the meter to the pH mode.
- 3. Turn the calib control until a 7.00 is displayed.
- Turn the temp/slope control (°C/slope) to the 25°C position.

The meter is now ready to calibrate the 97-08 Dissolved Oxygen Electrode. Refer to the instruction manual for additional information and for Table 1 and Appendices.

- Turn the mode switch on the electrode to BT CK. Good battery operation is indicated by a reading of 13.00 or greater on the meter.
- Turn the mode switch on the electrode to ZERO. Use the zero calibration control to set the meter to read 0.00.
- 3. Insert the funnel into a BOD sample bottle containing enough water to just cover the bottom. Insert the electrode, making sure that the electrode tip is not immersed in the water and does not have water droplets clinging to the outside of the membrane. Let stand approximately 30 minutes to ensure water saturation of air in BOD bottle. Also use this bottle for storage between measurements.
- 4. Turn the electrode mode switch to the AIR position. If measurements are being made at sea level, use the AIR calibration control to set the pH meter reading to the prevailing barometric pressure (divided by 100). If the barometric pressure is unknown, if the elevation is above sea level or if the sample has a salinity greater than 2 parts per thousand, consult **Table 1** found in the 97-08 instruction manual to obtain the correct AIR setting.
- Turn electrode mode switch to H₂O for sample analysis.

ORION

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APPENDIX E

Standard Operating Procedure for the YSI Specific Conductivity Meter Model 33

STANDARD OPERATING PROCEDURES FOR THE

YSI SPECIFIC CONDUCTIVITY METER MODEL 33

1 INTRODUCTION

This manual explains the use of the Model 33 Specific Conductivity Temperature (S-C-T) Meter. The instrument is designed for measurements of temperature in the range of -2 to 50° C, salinity in the range of 0 to 40%, and conductivity in the 0-50, 0-500 and 0-5000 mhos/cm with the YSI 3300 Series Probe.

2 QUICK REFERENCE OPERATION

- 1. Turn on instrument.
- 2. Adjust meter to zero by turning screw on meter face to coincide with the zero on the conductivity scale. Utilize wand and mirror to ensure accurate reading.
- 3. Turn MODE control to REDLINE and adjust REDLINE control so the meter needle lines up with REDLINE on meter face.
- 4. Plug probe into probe jack on the side of the instrument.
- 5. Place probe into sample.
- 6. Set MODE switch to TEMPERATURE.
- 7. Allow one minute for probe temperature to equilibrate.
- 8. Read temperature on bottom of scale in degrees Celsius.
- 9. Set MODE switch to appropriate setting, starting at the X100 scale and working down to the X1 scale.
- 10. Allow 30-40 seconds for needle to stabilize.
- 11. Take reading and multiply by the correct scale. The answer is expressed in mhos/cm.

Example: Meter Reading 238

Scale X 10 Answer: 2380 mhos/cm

12. Reset MODE to REDLINE.

3 THEORY OF OPERATION

Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions, their total concentrations, mobility, valence and relative concentrations, and on the temperature of measurement. Electrolytic conductivity increases with temperature at a rate of approximately 1.9% per degree centigrade (° C). Significant errors can result from inaccurate temperature measurement.

The conductivity of potable waters in the United States ranges generally from 50 to 1500 mhos/cm. The conductivity of domestic waste waters may be near that of the local water supply, although some industrial wastes have conductivities greater than 10,000 mhos/cm.

The physical measurement made in a laboratory determination of conductivity is usually of resistance, measured in ohms or megohms. The resistance of a conductor is inversely proportional to its cross-sectional area and directly proportional to its length. The magnitude of the resistance measured in an aqueous solution therefore depends on the characteristics of the conductivity cell used, and is not meaningful without knowledge of these characteristics. Specific resistance is the resistance of a cube, whose edges are one centimeter in length. Practical electrodes measure a given fraction of the specific resistance, the fraction being the cell constant, C:

 $C = Measured resistance, R_m$ Specific resistance, R_s

The reciprocal of resistance is conductance. It measures that ability to conduct a current and is expressed in reciprocal ohms or mhos. A more convenient unit in water analysis is micromhos. When the cell constant is known and applied, the measured conductance is converted to the specific conductance or conductivity, R_s, the reciprocal of the specific resistance:

$$\frac{1}{R_s} = \frac{C}{R_m}$$

The term "conductivity" is preferred and customarily is reported in micromhos per centimeter (mhos/cm). A conductance measurement requires a source of electrical power, a cell to contain the solution, and a suitable bridge to measure the resistance of the solution. The YSI utilizes two D-size alkaline batteries providing approximately 200 hours of operation. The cell

itself is a rugged plastic conductivity cell and a precision thermometer temperature sensor combined in a single unit with the bridge being transistorized. The bridge used is similar to the Wheatstone Bridge.

4 CALIBRATION

4.1 CALIBRATION FOR FIELD

- 1. Turn instrument on and connect probe.
- 2. Set ZERO by aligning needle to the ZERO mark and adjusting the screw on meter face. Do this gently so instrument is not damaged.
- 3. Switch MODE to REDLINE. The needle should line up with the REDLINE that is approximately 2/3 across the meter. Adjust if needed by using REDLINE knob.

4.2 IN-HOUSE CALIBRATION

1. Read the temperature and conductivity of a 0.01 normal KCl solution. Determine the salinity of the solution by running a line vertically on a graph from this conductance value until it intersects the appropriate ° C line (interpolate as required for temperature between the given ° C lines). From this intersection, extend a line horizontally to the edge of the graph. This determines the salinity for this sample.

Example: 25,000 mhos/cm and 20° C gives a salinity of 17. Example: 2,500 mS/m and 20° C gives a salinity of 17.

- 2. Remove the °C knob, switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in Step 1. In the example given, the value is 17.
- 3 Switch to TEMPERATURE. (Note: This temperature reading must be the same as Step 1.) Place the knob on the control shaft (without turning the control shaft), with the knob pointer at the same temperature as the meter reading and tighten both set screws securely. The temperature knob may become loose or slip

from its normal position. In an emergency, the dial can be re-positioned. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

4.3 CELL CALIBRATION IN-HOUSE

The YSI #3300 Series Cells are calibrated to absolute accuracy of +/- 1.5% based on a standard solution. Since the literature on conductivity does not indicate a consistently accepted standardization method, Burlington has chosen the 0.01 normal KCl solution method as determined by Jones and Bradshaw in 1937. The solution is prepared by diluting 0.745 grams of pure dry KCl with distilled water until the solution weighs 1.00 kilograms. The table below shows the values of conductivity this solution would have if the distilled water were non-conductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

TEMPERATURE (°C)	CONDUCTIVITY (mhos/cm)
15	1141.5
16	1167.5
17	1193.6
18	1219.9
19	1246.4
20	1273.0
21	1299.7
22	1236.6
23	1353.6
24	1380.8
25	1408.1
26	1436.5
27	1463.2
28	1490.9
29	1518.7
30	1546.7

The operator may use the standard solution and the table to check accuracy of a cell's constant or to determine an unknown constant. The formula is shown below:

$$K = \frac{R(C_1 + C_2)}{10^6} \text{ or } \frac{R(S_1 + S_2)}{10^5}$$

Where: K = Cell Constant

R = Measured resistance in ohms

 $C_1 = Conductivity in \mu mhos/cm$

 \tilde{C}_2 = Conductivity of the distilled water, in μ mhos/cm

 $S_1 = Conductivity in mS/m$

S₂ = Conductivity of the distilled water used to make the solution, in mS/m

R, C_1 , and C_2 , or S_1 and S_2 , must either be determined at the sample temperature or corrected to the same temperature to make the equation valid.

NOTE: For further information on conductivity and the above standard information, refer to ASTM Standards Part 23: Standard Methods of Test for Electrical Conductivity, or Water and Industrial Waste Water - ASTM Designation D1125-64.

5 DETAILED FIELD OPERATION AND SETUP PROCEDURES

SETUP:

- 1. Adjust meter to zero by turning bakelite screw on meter face to coincide with zero on conductivity scale.
- 2. Calibrate meter by turning on MODE switch to REDLINE and adjusting the REDLINE control so the meter needle lines up with REDLINE on meter face.
- 3. Plug probe into probe jack in side of instrument.
- 4. Rinse probe twice with distilled water.
- 5. Place probe in sample, making sure probe is completely submerged, at least one to two inches.

- 6. Switch MODE control to TEMPERATURE.
- 7. Let stabilize (approximately 30 to 60 seconds) and take reading on bottom scale.
- 8. Switch the MODE control to conductivity on the X100 scale. If the reading falls below 50 on the 0-500 scale, switch to X10 scale. Let stabilize (30-60 seconds), take reading and multiply by the meter scale used. Answer is expressed in mhos/cm.

Example meter reading: 137

Scale:

X100

Answer:

13,700 mhos/cm

- 9. Reset MODE to REDLINE.
- 10. Remove probe from sample and rinse twice thoroughly with distilled water.
- 11. Keep probe in a container filled with distilled water between samples. This is extremely important since the probe may overheat on hot days or cause heating of the sample when a temperature measurement is being made.

6 FIELD QA/QC

QA/QC considerations involve replicate measurements, time for equilibrium, collection to measurement time, redline, temperature check, and conductivity QA/QC controls. These are outlined below.

Measurement of temperature and conductivity should be made as soon after collection of sample as possible. The recommended method is to use a four-liter container (properly cleaned plastic will do), 3/4 filled after rinsing twice with solution to be sampled. Rinse probe twice with distilled water and insert in sample submersing probe at least 1 to 2 inches, swirl gently. When taking measurements, allow the instrument to stabilize. The time required for equilibration is usually 45-60 seconds. Conductivity should be taken immediately after the temperature reading. The results are to be recorded in the field logbook in tabular form on a Field Data Record.

Standard Field Practice:

- 1. A check on redline measurements must be made before probe is inserted in sample. This should be easy to do, since it should be in the redline mode between samples.
- 2. A check on the temperature should be made at least once per hour of use. This could be done using a mercury thermometer and comparing the two readings. They should agree to within 0.5°C.
- 3. Duplicate measurements must be made at a rate of one per ten samples. To do this, follow instructions for sample measurements. After recording results, remove probe, rinse twice, immerse probe into same sample and take reading again. The result should differ from the previous measurement by 1 percent or less. Log all results on proper form or logbook.
- 4. Split sample measurements can be substituted for duplicate measurements. For split measurements, divide the sample in half, and take reading for each half. The two results should differ by 1 percent or less. Log results in field book.

7 MAINTENANCE

7.1 FIELD MAINTENANCE

The only field maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready D95 or equivalent, will provide approximately 200 hours of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished. To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The positive (+ button) end must go on red.

Once every six months, or when the probe indicates low readings, wash the electrodes in soapy lukewarm water, and rinse with distilled water. Then soak overnight in distilled water. Be careful not to touch electrodes on the inside or damage to the probe can result.

7.2 IN-HOUSE MAINTENANCE

CLEANING

When the cell test indicates low readings, the probable cause is dirty electrodes. Hard water deposits, oils and organic matter are the most likely contaminants.

For normal cleaning, soak the electrodes for five minutes with a locally available bathroom tile cleaning preparation such as Dow Chemical "Bathroom Cleaner", Horizon Industries "Rally, Tile, Porcelain, and Chrome Cleaner", Johnson Wax "Envy, Instant Cleaner", or Lysol Brand "Basin, Tub Tile Cleaner".

For stronger cleaning, a five-minute soak in a solution made of ten parts distilled water, ten parts isopropyl alcohol and one part HCl can be used. Always rinse the probe after cleaning and before storage.

CAUTION: Do not touch the electrode inside the probe. Platinum black is soft and can be scraped off. If cleaning does not restore the probe performance, replatinizing is required.

REPLATINIZING

Equipment required:

- YSI #3140 Platinizing Solution, 2 fluid ounces (3% platinum chloride dissolved in 0.025% lead acetate solution);
- YSI Model 33 or 33M S-C-T Meter;
- 50 ml glass beaker or equivalent bottle; and
- distilled water.

Procedure:

- 1. Clean the probe as described above.
- 2. Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes. Do not cover the top of the probe.
- 3. Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrode. Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate times shown below.

Meter Reading	Time
(mhos/cm)	(Minutes)
30,000	5
25,000	6
20,000	8
15,000	11
10,000	16

- 4. After the elapsed time, remove the probe and rinse in fresh water.
- 5. Return the solution to its container. Two ounces of solution should be sufficient for 50 treatments.

PROBE USE

- 1. Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as pliers or weights should be kept at least six inches from the probe.
- 2. Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 weights are supplied in pairs with a total weight of four ounces per pair. If it becomes necessary to add more weight to overcome currents, Burlington suggests limiting the total weight to two pounds (eight pairs). For weights in excess of two pounds, use an independent suspension cable. In either case, weights must be kept at least six inches away from the probe.

3. Gentle agitation by raising and lowering the probe several times during an in situ measurement causes flow of sample solution through the probe and improves the time response of the temperature sensor.

8 STORAGE

In the field, as well as in-house, it is best to store conductivity probes in deionized water between measurement. A probe stored in water requires less frequent platinization.

Store the instrument in its carrying case, making sure the straps are in place and the probe is locked down. If the instrument and probe are packed separately, store the probe in a plastic bag. Ensure that the probe is securely packed in order to avoid accidental knocks and possible damage.

Each day in use, wipe down instrument and clean with water before bringing back to be stored.

APPENDIX F

RECON® System Standard Operating Procedures

STATEMENT OF PROCEDURES

RECON MULTIMEDIA SAMPLING AND TESTING SYSTEM

August 1992

RECON MULTIMEDIA SAMPLING AND TESTING SYSTEM PROCEDURES

With the Burlington RECON System, various media are sampled and tested for an array of volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs). These tests are designed to detect the presence, potential magnitude and extent of contaminants in the subsurface. The sample collection and chemical analysis equipment are fully contained within a one-ton van. The Burlington RECON System can be used for a variety of investigative purposes, including:

- identifying contaminant sources;
- defining contaminant plumes;
- measuring product thickness in groundwater;
- preparing vertical profiles of contamination in soil;
- monitoring landfills for accumulations of soil-gas;
- testing for leaks from underground storage tanks (USTs);
- identifying potential hazards during site assessments and environmental audits; and
- screening for contamination prior to drilling and well installation.

HOW THE BURLINGTON SYSTEM WORKS

Based on historical and current site conditions and other factors, Burlington technicians select a sample location and hydraulically drive a probe to the desired sampling point. The RECON System pneumatic probe unit is capable of delivering more than 15,000 pounds of downward force. This unit can drive a probe up to 35 feet deep and can drill through as much as 30 inches of concrete or asphalt at the surface.

Depending on the complexity of the site and the media tested, the RECON System can be used to sample and analyze compounds at 10 to 25 locations per day.

Burlington technicians collect soil-gas, groundwater, free product, or soil samples and use a laboratory-grade gas chromatograph (GC) mounted in the RECON van for analysis. With the appropriate detectors and columns, the GC can measure and analyze:

- volatile priority pollutants;
- petroleum hydrocarbons;
- industrial solvents;
- polychlorinated biphenyls (PCBs); and
- pesticides and herbicides.

SOIL-GAS SAMPLING AND ANALYSIS PROCEDURES

Soil-gas samples are obtained by inserting a 0.75-inch outside diameter (O.D.) steel probe to a predetermined depth. The *in situ* vapor sample is withdrawn through the probe and captured in an air-tight gas sampling bulb. The probe hole is grouted, and if needed, the asphalt or concrete is patched.

For a standard soil-gas investigation, a vertical profile at a single probe hole location is performed to identify the appropriate depth where samples can be collected to obtain optimum results.

After sample collection using the probe, the sampling bulb is marked for identification and removed from the sampling system. A portion of the soil-gas sample is withdrawn from the sampling bulb with a gas-tight syringe and injected into the GC for analysis. All of the soil-gas samples collected are analyzed using a flame-ionization detector (FID) and a Volatile Organic Compound (VOC) capillary column. The results of the analysis will be automatically recorded

in the computer, which is directly linked to the GC. The analytical data is stored and available for on-site recall.

Quality Assurance/Quality Control (QA/QC) guidelines are maintained so the data collected from the survey are accurate and reliable. These QA/QC samples consist of duplicate samples of *in situ* soil-gas, chromatographic equipment blank samples, equipment blanks obtained from regularly decontaminated sample probes and bulbs, ambient air samples, and calibration standards. Field QC samples are collected during each day of field activity.

Prior to use in sample collection, all sampling equipment, probe rods, and sample bulbs are decontaminated by pressure washing and purging. Pressure cleaning and detergent washing are used to decontaminate all tools and equipment. Further decontamination procedures are outlined in the site-specific sampling plan.

The soil-gas survey data are presented in a final report summarizing the sampling and analysis of target compound vapors in the vadose zone. The presence of detectable levels of target compounds is dependent upon several factors, including the permeability of soils, the depth to groundwater, and whether sufficient concentrations of target compounds are present in the aqueous phase to facilitate volatilization into the vadose zone.

GROUNDWATER SAMPLING AND ANALYSIS PROCEDURES

Groundwater sampling using the Burlington RECON System is a method of rapid screening to establish approximate levels of contamination. Data collected by this method is often used to support and enhance conventional groundwater monitoring programs.

Groundwater grab samples are obtained by inserting a 0.75-inch O.D. steel probe to a depth that intercepts the groundwater table. Samples are collected by one of the three methods for groundwater grab sampling described below. The method used depends on several factors, including decontamination requirements, efficient use of time and materials, analytical requirements, and the depth to groundwater. Samples can be collected by pump or a miniature

stainless-steel bailer. If a pump is used, either a peristaltic or vacuum pump is used to draw the sample to the surface through polyethylene tubing. After sample collection, the probe hole is grouted, and if needed, the asphalt or concrete is patched.

The groundwater samples are collected in glass vials and analyzed on site by Burlington. Samples are analyzed with a laboratory-grade GC using a FID and modifications to U.S. Environmental Protection Agency (EPA) SW-846 Method 8010/8020. The modifications include the use of an FID in the GC, a VOC capillary column, static headspace analysis, single-point calibration standard, and no sample spikes. The sample is placed into a glass vial fitted with a septum cap and heated. This procedure partitions the VOCs between the sample and headspace in the vial. A sample aliquot is removed from the vial with a gas-tight syringe and injected directly into the GC.

QA/QC guidelines are maintained so the data collected from the survey are accurate and reliable. These QA/QC samples consist of duplicate samples of *in situ* groundwater, chromatographic equipment blank samples obtained from regularly decontaminated sample probes, and calibration standards. Field QC samples are collected during each day of field activity.

Prior to use in sample collection, all sampling equipment and probe rods are decontaminated by pressure washing and purging. Pressure cleaning and detergent washing are used to decontaminate all tools and equipment. Further decontamination procedures are outlined in the site-specific sampling plan.

The groundwater survey data are presented in a final report summarizing the sampling and analysis of target compound vapors in the groundwater. The presence of detectable levels of target compounds is dependent upon several factors, including the permeability of soils, the depth to groundwater, and whether sufficient concentrations of target compounds are present in the aqueous phase to facilitate sample volatilization.

SUBSURFACE SOIL SAMPLING AND ANALYSIS PROCEDURES

Subsurface soil samples are obtained by inserting a 0.75-inch O.D. steel probe equipped with a retractable drive-point sampler to the top of the desired sampling depth. The sampler is then advanced a minimum of 12 inches to impact soil into the sampling sleeve. After sample collection, the probe hole is grouted, and if needed, the asphalt or concrete is patched.

Samples are extruded into a glass vial and analyzed on-site by Burlington. Samples are analyzed with a laboratory-grade GC using a FID and modifications to EPA SW-846 Method 8010/8020. The modifications include the use of an FID, a VOC capillary column, static headspace analysis, a single-point calibration standard, and no sample spikes. The sample is placed into a glass vial fitted with a septum cap. A sodium sulfate-saturated solution is added to the vial, and the vial shaken to disperse the soil. The vial is then heated. This procedure partitions the volatile compounds between the saturated solution and headspace in the vial. A sample aliquot is removed from the vial with a gas-tight syringe and injected directly into the GC.

QA/QC guidelines are maintained so the data collected from this survey are accurate and reliable. These QA/QC samples will consist of duplicate samples of soil, chromatographic equipment blank samples, equipment blanks obtained from regularly decontaminated sample probes, and calibration standards. Field QC samples are collected during each day of field activity.

Prior to use in sample collection, all sampling equipment and probe rods are decontaminated by pressure washing and purging. Pressure cleaning and detergent washing are used to decontaminate all tools and equipment. Further decontamination procedures are outlined in the site-specific sampling plan.

The data are presented in a final report summarizing sampling and analysis of target compounds in the soil. The presence of detectable concentrations of target compounds is dependent upon several factors, such as soil type. If the soils are potentially impacted from groundwater or vapors emanating from contaminated groundwater, the presence of detectable

concentrations is also dependent upon the permeability of soils, the depth to groundwater, and whether sufficient concentrations of target compounds are present in the aqueous phase to facilitate volatilization.

PIEZOMETER INSTALLATION PROCEDURES

The use of piezometers is a rapid method to evaluate product thickness and groundwater gradients. Groundwater sampling with piezometers is a method to rapidly screen an area for approximate levels of contamination. Data collected by this method is often used to support and enhance conventional groundwater monitoring programs.

The piezometers consist of threaded one-inch-diameter polyvinyl chloride (PVC) 0.01-inch slotted screen and riser pipe. The hydraulic probe unit is used to drive the piezometer assemblies, which consist of three-foot lengths of 0.75-inch-diameter threaded steel inner rods and the PVC piezometer sections. The piezometers are installed by pushing the piezometer sections simultaneously with the inner rods. A hydraulic hammer is used to assist in driving the assemblies through unusually hard soil. The inner rods are withdrawn using the hydraulic probe unit after the piezometers have been set to the desired length.

SITE ACCESS, HEALTH AND SAFETY, AND UTILITY CLEARANCE

Site conditions are expected to permit the work to be performed utilizing a two-wheel-drive van. Provisions for road building or towing the van normally are not included in the work scope, although adjustments can be made as necessary.

Our personnel will comply with Burlington's and the client's health and safety requirements on site as specified in the site-specific Health and Safety Plan. Air-purifying respirators are used if personnel exposures could exceed the Occupational Safety and Health

Administration (OSHA) permissible exposure limits specified in 29 CFR 1910.1000 (revised January 19, 1989). All Burlington employees assigned to the project will have been trained to use air-purifying respirators in accordance with OSHA regulations, will have passed a respirator fit test, and are participating in a medical monitoring program. Respirators and disposable personal protective clothing (Level C or B) are available if necessary.

Unless specific arrangements are made otherwise, the client will provide all utility clearances prior to investigation efforts. Availability of an on-site water source (10 gallons per minute) and an on-site location for decontamination of RECON equipment will be provided by the client.

QUALITY CONTROL

QC is an essential part of an analytical test methodology. QC procedures increase the confidence in the analytical results and are used to evaluate the reproducibility of the data.

The GC used to analyze samples is calibrated using a known concentration of each of the target compounds of interest at the beginning of the day before the analysis of any samples. The EPA recommends instrument calibration be performed at least once every 12 hours. The calibration helps to evaluate the operating conditions of the GC.

A chromatographic system blank is analyzed every 10 samples as a means of indicting that sample carryover has not occurred. If sample carryover has occurred, the concentration detected in the system blank can be subtracted from any of the subsequent samples containing that compound. A probe rod blank is analyzed prior to sample collection to ensure that rods are free of contamination.

A duplicate sample is analyzed once every 10 samples, or at least once daily for each survey. Burlington's standard operating procedure (SOP) for analyzing samples using a static headspace method is in Attachment A of this Statement of Procedures.

HEALTH AND SAFETY GUIDELINES

Burlington's Health and Safety SOP for RECON investigations is provided in the site-specific Health and Safety Plan. Procedures for working in other site conditions, employee requirements, and chemical safety protocol are also outlined in the site-specific Health and Safety Plan.

ATTACHMENT A

RECON System Field Screening for Volatile Organic Compounds
Using Static Headspace Method

BURLINGTON ENVIRONMENTAL

RECON® SYSTEM FIELD SCREENING FOR VOLATILE ORGANIC COMPOUNDS USING STATIC HEADSPACE METHOD

Scope and Application

This method is for the evaluation of volatile organic compounds in soil-gas, groundwater, and soil.

Method

Headspace-gas chromatography using megabore capillary columns and flame-ionization detection (FID)

Reference

"EPA Test Methods for Evaluating Solid Waste," SW-846 Methods 3810, 8010, and 8020 with modifications

Lower Quantifiable Limits (LQL)

Headspace 1.0-10.0 micrograms per liter ($\mu g/L$) or less (compound specific)

Sample Handling

Water and soil samples are to be collected in 40-milliliter (mL) vials with open screw-caps and Teflon-faced silicon septa. Water samples should be collected so that no headspace remains in the bottle. Soil-gas samples are to be collected in 250-mL glass bulbs. Sample should be collected in a manner to ensure the complete purging of the bulb. All samples should be protected from sunlight and transported to the field laboratory as soon as possible. Water samples will be held on ice prior to analysis.

Reagents and Apparatus

 Open screw cap 40-mL vial (I-CHEM). Detergent washed, distilled water rinsed and dried at 105°C before use.

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- Septum Teflon-faced silicon (I-CHEM). Detergent washed, distilled water rinsed and dried at 105°C before use.
- 250-mL gas sampling bulbs (Teflon stop-cocks and septum inlet).
- 4. Gas chromatograph Hewlett-Packard (HP) Model 5890A equipped with an FID.
- 5. Computing integrator HP Model 3396 A/B and dual-disk drive storage device.
- 6. Column 1 SE-54 wide-bore capillary column, 0.53 mm i.d., 15 meter, 1.2-micron phase.
- 7. Syringes Assorted glass gas-tight microliter syringes and liquid syringes (10-1000 uL volumes).
- 8. Balance $-\pm$ 0.0001 grams (g) Sartorious Analytical Balance (not on board RECON System Van).
- 9. Reagent water organic free water or distilled-deionized water that has been shown to be organic-free at the method detection limit.
- 10. Constant temperature heat block = 70°C (capable of ± 0.5°C temperature control).
- 11. Volumetric flasks assorted.
- 12. Pipettes assorted.
- 13. GC operating materials and supplies as described below "Chromatographic Conditions".

Calibration Standard Preparation

- 1. Stock standard solutions: Prepare a VOC standard at 2,500 micrograms per milliliter ($\mu g/mL$) in methanol.
 - a. Add about 30 mL of methanol to a 50-mL volumetric flask. Allow the flask to stand unstoppered until the methanol on the neck of the flask has dried.
 - b. Tare the flask on the analytical balance.
 - c. Using a 100- μ L syringe, add 0.125 g (correct for percent purity) of the reference material to the flask. Make

sure the drops fall directly into the methanol without contacting the neck of the flask.

- d. Determine the mass of reference material added. Rinse the syringe with methanol, tare the flask, and add the next standard.
- e. After all the reference materials are added, fill to volume with methanol, cap, and invert to mix.
- f. Transfer stock standard solution to 40-mL VOA vials, cap, and store in a cool, dark location.

Calibration - Vapor Standard - Single Point Calibration Procedure

- 1. Prepare a vapor standard using a nonapproved modification to USEPA Method TO-1, calibration standard preparation using a static dilution bottle technique. Add appropriate amount of stock standard to a known volume vessel, fitted with a Mininert® valve and heat to 70°C (at ± 0.5°C). Inject aliquot directly into the gas chromatograph.
- 2. Use a single-point standard curve of peak area response versus total concentration injected for each of the compounds of interest.
- 3. A continuing calibration check is performed after each set of 20 samples and as the last sample of the day. If the response for any of the compounds varies from the expected response by more than ±30 percent, the average response should be used.

Sample Analysis

Soil-Gas Samples:

- 1. Soil gas samples are received in 250-mL glass bulbs. When received, they are allowed to equilibrate to the ambient air temperature.
- 2. Remove an aliquot of the sample and inject directly into the gas chromatograph.
- 3. If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller aliquot is taken from the same sample bulb.

Water Samples:

- 1. Water samples are received in 40-mL VOA vials. Uncap and remove a 10-mL aliquot of the sample from the vial. Dispense the sample into a headspace vial.
- 2. The vials are placed in a 70°C aluminum heat block and heated for 5 minutes.
- 3. Remove an aliquot of headspace and inject directly into the gas chromatograph.
- 4. If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller sample volume is injected into the instrument and the analysis repeated.

Soil Samples:

- 1. Soil samples are received in 40-mL VOA vials. A 10-gram quantity of soil is placed in a clean VOA vial.
- 2. Add 10-mL of a sodium sulfate saturated solution to the vial.
- 3. Shake the vial vigorously to disperse the soil.
- 4. Place the vial in a 70°C heat block and heat for five minutes.
- 5. Remove an aliquot of headspace and inject directly into the gas chromatograph.
- 6. If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller sample volume is injected into the instrument and the analysis repeated.

Chromatographic Conditions:

Column: 15-meter J&W SE-54 wide-bore capillary column,

0.53 mm i.d./1.2-micron phase

Carrier Gas: Hydrogen - Ultra High Purity Grade 8 mL/min

Detector: Flame-ionization detector (FID)

Detector Temperature: 300°C

Hydrogen Flow: 35 mL/min

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Zero Air Flow:

450 mL/min

Nitrogen Make-up Gas: Hydrogen Carrier flow: 8 mL/min

20-25 mL/min

Injector Temperature:

200°C

Oven Temperature

Profile:

Initial: Rate: 45°C - 0.5 min.

20°C/min.

Final

220°C - 5 min.

Conditions listed can be varied as needed for changing applications.

Analytical Calculations

All calculations will be automatically performed by the computerized integrator.

- Review the chromatograms and data reports for analysis. Check for gross errors such as incomplete data reports because of faulty integration.
- Prepare external standard calibration curves for each compound 2. using a single point calibration curve. Calculate the lower quantifiable limit (LQL) for each target compound.
- Calculate the concentration found in the samples from the 3. calibration curves using the following equation:

ug = Area Comp x RF Target x DF

in which:

DF

= Dilution Factor

RF

= Response Factor (Target Compound)

Area Comp = Area of compound in sample.

Data Reporting

- All results, chromatographic parameters, analytical results, sampling locations, and notes will be recorded in daily GC field worksheets.
- All data generated by the field GC will be considered as 2. tentatively identified, with concentrations being estimated.

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3. All raw field data will be forwarded to the Mathes office for final review and QA/QC prior to final reporting.

Quality Control

- 1. Each daily analytical run should begin with a GC system blank and a headspace standard calibration. Calibration retention time check standards should be analyzed every 10 samples. A continuing calibration check is performed after eachs et of 20 samples and as the last sample of the day. Continuing calibration standards should be within 30 percent of the original standards or a new standard curve should be prepared and samples analyzed since the last check standard reanalyzed.
- 2. A minimum of 10 percent duplicate samples should be analyzed or a minimum of one per day. Duplicates should be within ±25 percent.
- 3. Gas chromatograph sample blanks will be analyzed at least one every 10 samples, or a minimum of one per day.
- 4. New stock standards should be prepared monthly in the laboratory. New working standards should be prepared daily.

APPENDIX G

Project Field Audit Checklist

FIELD AUDIT CHECKLIST Briefing with Site Manager

DATE OF AUDIT SIGNATURE OF AUDITOR
QA Project Plan and a Site Health and Plan plan prepared? If yes, what items dressed in the plan? ts:
briefing held with project participants?
dditional instructions given to project ipants (i.e., changes in project plan)?
re a written list of sampling locations scriptions?
re a map of sampling locations?

Yes No N/A 6.	Does the sampling team follow a system of accountable documents?
	If yes, what documents are accountable?
	Comments:
Yes No N/A 7.	Is there a list of accountable field documents
	checked out to the site manager or designated
	person?
	If yes, who checked them out?
	Comments:
Yes No N/A 8.	Is the transfer of field documents (Traffic
	Reports, Chain-of-Custody Records, logbooks,
	etc.) from the site manager to the field
	participants documented in a logbook?
	Comments:

FIELD AUDIT CHECKLIST Field Observations

PROJECT NO.	DATE OF AUDIT
PROJECT MANAGER	SIGNATURE OF AUDITOR
	Was permission granted to enter and inspect
	the facility?
	Comments:
Yes No N/A 2.	Is permission to enter the facility documented?
	Comments:
Yes No N/A 3.	Were split samples offered to the facility?
	If yes, was the offer accepted or declined?
	Comments:
Yes No N/A 4.	If the offer to split samples was accepted,
	were the split samples collected?
	Comments:
Yes No $\frac{N/A}{}$ 5.	Is the offering of split samples recorded?
	Comments:

Yes	No	N/A	6.	Îf split samples were collected, are they
				documented?
				If yes, where are they documented?
				Comments:
Yes	No	N/A	7.	Are the number, frequency, and types of field
			• •	measurements and observations taken as speci-
				fied in the project plan or as directed by
				the site manager?
				Comments:
	•			
Yes	_ No _	N/A	8.	Are field measurements recorded (pH, tempera-
				ture, conductivity, etc.)? Where?
				Comments:
Yes	No	N/A	9.	Are samples collected in the types of containers
				specified in the project plan or as directed by
				the site manager?
				Comments:
V ~ ~	No	NI / R	10	Are samples preserved as specified in the Project
169	140	. 14/2-	10.	·
				Plan or as directed by the site manager?
				Comments:

Yes <u> </u> No _.	_ N/A	11.	Are the number, frequency, and types of samples collected as specified in the Project Plan or as directed by the site manager? Comments:
Yes No .	N/A	12.	Are samples packed for preservation as specified in the Project Plan (i.e., packed in ice, etc.)? Comments:
Yes No .	_ N/A	13.	Is sample custody maintained at all times? Comments:

FIELD AUDIT CHECKLIST Document Control

DATE OF AUDIT
SIGNATURE OF AUDITOR
Have all unused and voided accountable documents been returned to the site manager by the team members? Comments:
Have document numbers of all lost or destroyed accountable documents been recorded in the site manager's logbook? Comments:
Are all samples identified with Traffic Report Stickers? Comments:
Are all Traffic Reports completed (e.g., station no., location, date, time, analyses, signatures of samplers, type, preservatives, etc.)?

Yes No N/A 5.	Are all samples collected listed on a Chain- of-Custody Record? If yes, describe the type of Chain-of-Custody Record used. Comments:
Yes No N/A 6.	Are the Traffic Report Sample Sticker numbers recorded on the Chain-of-Custody Records? Comments:
Yes No N/A 7.	Does information on Traffic Reports. Tags and Chain-of-Custody Records match? Comments:
Yes No N/A 8.	Do the Chain-of-Custody Records indicate the method of sample shipment? Comments:
Yes No N/A 9.	Is a Chain-of-Custody record included with the samples in the shipping container? Comments:
Yes No N/A 10.	If used, are blank samples identified? Comments:

Yes	No _	N/A	11.	If collected, are duplicate samples identified on Traffic Reports and Chain-of-Custody Records? Comments:
Yes	No _	N/A	12.	If used, are spiked samples identified? Comments:
Yes	No _	_ n/a	13.	Are Field Notebooks signed by the individual who checked out the notebook from the site manager? Comments:
Yes	No _	_ N/A	14.	Are Field Notebooks dated upon receipt from the site manager? Comments:
Yes	No _	_ N/A	15.	Are Field Notebooks project-specific (by notebook or by page)? Comments:
Yes	No _	_ N/A	16.	Are Field Notebook entries dated and identified by author? Comments:

Yes	No	N/A	17.	Is the facility's approval or disapproval to
				take photographs noted in a Field Notebook?
				Comments:
Yes	_ No _	. N/A	18.	Are photographs documented in Field Notebooks
				(e.g., time, date, description of subject,
				photographer, etc.)?
				Comments:
			4.0	
Yes	_ No _	N/A	19.	If a Polaroid camera is used, are photos matched
				with Field Notebook documentation?
				Comments:
Yes	No	N/A	20.	Are Traffic Report numbers recorded in the
100			20.	
				site manager logbook?
				Comments:
Yes	No	N/A	21.	Are Quality Control checks documented (i.e.,
				calibration of pH meters, conductivity meters,
				etc.)?
				Comments:

Yes No N/A	22.	Are amendments to the Project Plan documented
		(on the Project Plan itself, in a project
		logbook, elsewhere)?
		Comments:

FIELD AUDIT CHECKLIST Debriefing with Site Manager or Field Sampling Team Leader

PROJECT NO.	DATE OF AUDIT
PROJECT MANAGER	SIGNATURE OF AUDITOR
OFFICE LOCATION	
Yes No N/A 1	. Was a debriefing held with project partici= pants after the audit was completed? Comments:
Yes No N/A 2.	. Were any recommendations made to project participants during the debriefing? If yes, briefly describe what recommendations were made. Comments: